### Commonwealth of Pennsylvania.

# DEPARTMENT OF AGRICULTURE, DAIRY AND FOOD DIVISION

**BULLETIN No. 224** 

# COMMERCIAL TABLE SIRUPS AND MOLASSES

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N. B. CRITCHFIELD, Secretary of Agriculture

JAMES FOUST, Dairy and Food Commissioner

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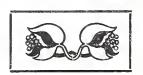


### PREFACE

In view of the great and growing consumption of Commercial Table Sirups and Molasses by the people of this country, and especially by children, the question of the wholesomeness of these products is of interest to every citizen. The following report, covering a wide range of sirups and molasses, such as are found in almost every family, is, therefore, made, and authorized to be printed as a Bulletin of this Department for the information of the Public.

N. B. CRITCHFIELD,

 $Secretary\ of\ Agriculture.$ 



### LETTER OF TRANSMITTAL

Harrisburg, Pa., March 30, 1912.

Hon. N. B. Critchfield, Secretary of Agriculture, Harrisburg, Pa.

Dear Sir: I have the honor to transmit herewith a report by Dr. William Frear, Chemist of this Bureau, upon the commercial table sirups and molasses on the markets of this Commonwealth, with the recommendation that it be published as Bulletin No. 224 of the Department.

Very respectfully,

JAMES FOUST,
Dairy and Food Commissioner.



State College, Pa., March 30, 1912.

Hon. James Foust,
Dairy and Food Commissioner,
Harrisburg, Pa.

Dear Sir: I have the honor to submit herewith my report upon the analyses of commercial table sirups and molasses submitted for investigation by the official sampling agents of the Dairy and Food Bureau.

Very respectfully,

WILLIAM FREAR,

Vice-Director and Chemist, The Pennsylvania State College Agricultural Experiment Station.



## TABLE OF CONTENTS

American use of table sirups,	11
American use of table sit ups,	11
Principal constituents,	19
Maple sirups,	-
Sugar-cane products,	49
Sugar-cane products,	52
Cane sirup,	54
Molasses,	
Refined molasses,	57
Glucose or corn sirup,	71
Glucose of colli situp,	77
Corn sirups and Glucose compounds,	•
Summary,	90
Index,	93
Index,	



## A STUDY OF THE TABLE SIRUPS, MOLASSES, AND GLU-COSES (CORN SIRUPS) ON SALE IN PENNSYLVANIA.

### By WILLIAM FREAR

The food articles which are the subject of the present study form a very considerable element in the dietary of the average Pennsylvania family. While sugary liquids and sauces or sweet fruit preparations are common accompaniments of hot or batter-cakes and fritters, and jellies garnish the service with fowl and game in fashionable menus, the sirup jug and preserve jar or fruit-butter dish are part of the ordinary table furnishing for each meal in the every day living of the Pennsylvania family. In addition to butter, it is a very narrowly straitened family that does not also supply with its bread what is aptly termed, in the vernacular of the central counties, a "second spread" of sirup or jam, to be liberally applied to the buttered bread.

The commercial products, which together with numerous domestic preparations, serve these purposes are chiefly maple sirup, refinery sirup, cane molasses—very generally termed "New Orleans molasses," whatever its geographical origin—glucose, or "corn sirup," and much more rarely, cane sirup, and especially, various mixtures of these simple products. Sorghum sirup has practically disappeared, both as a domestic and a commercial article. Beet molasses, owing to its unpleasant flavor and odor, is not fit for table or kitchen use; whereas, cane molasses enters into many articles of cookery which their sugars sweeten, their flavors affect, and their acids serve to leaven, because these acids liberate rapidly the carbonic acid gas from the baking soda used as an associate ingredient.

### PRINCIPAL CONSTITUENTS

While each of these articles, except glucose or corn sirup, has its own distinctive flavor, due to various acids and to other flavoring principles not yet isolated and determined, all, with the exception just mentioned, have a sweet flavor due especially to cane-sugar and related sugars. Glucose, or corn sirup, a chemical product from starch, differs markedly in composition from the other articles under consideration, but is composed of substances belonging to the great group of carbohydrates, to which cane sugar and the other sugars

related thereto belong. Because these sugars belong in common to all the sirups proper and to molasses, their chemical and food characters will be briefly considered in these introductory paragraphs.

The carbohydrates are a great group of substances, including the cellulose, starch, gums and mucilages, dextrins and sugars of the plant world and the glycogen of the animal body. They are composed of the three elements, carbon, hydrogen, and oxygen, the two last named being present in the proportions in which they unite to form water; whence the group name, carbo-hydrates. They differ, one from the other, in chemical complexity, and are very largely convertible, one into the other, under the influences in the living plant cells, and, in far more limited degree, in living animal cells. By various laboratory processes, the chemist has been able to reproduce certain of these transformations, especially such as result in a change from complex to simple bodies; but, while recent years have witnessed marvellous advances in his art, he has not yet succeeded in building up from the simplest materials the more complex of these bodies. As food components, the carbohydrates serve as sources of energy and of fat, but not as building materials for the principal tissues of the body.

Of the carbohydrates, certain sugars and dextrins are of immediate interest in this connection. Of the sugars, two classes are represented: The mono-saccharoses or simple sugars, here including dextrose, levulose, and their mixture, invert sugar; and the di-saccharoses, here including cane sugar, (sucrose or saccharose), and maltose or malt-sugar. Of the dextrins, the achro-dextrins are of principal interest in this relation.

Before describing the properties of these several substances, it should be noted that they all belong to what are known as optically active bodies. If a clear crystal of Iceland spar (transparent crystaline carbonate of lime) be placed upon a printed page, the eye of the reader beholds a double image of each letter visible through the This is due to the fact that the spar is doubly refractive and breaks the light rays each into two parts. If crystals of Iceland spar be properly shaped and united by a transparent film of Canadian balsam, one of these two rays is totally reflected while the other passes through the united crystals which, thus combined, are known under the name of nicol-prism. If two such prisms be placed at the opposite ends of a metal tube and in like position with respect to the passing rays of light, the portion of light which has passed through the first of the prisms will pass through the second prism with little diminution of intensity; but if the second prism be turned through an angle of 90°, the light from the first prism is entirely cut off, being totally reflected as its twin ray was in passing the first prism. Because of this peculiar behavior of light which has passed through a nicol prism, the light is said to be polarized.

A polariscope is an instrument composed of a metal tube containing two nicol prisms; the first, at the end of the tube through which light enters, is called the polarizer; the second, mounted at the farther end of the tube in a ring which can be rotated, is called the analyzer. If the analyzer and polarizer of a polariscope be set in such position that the analyzer totally reflects polarized light which has passed through the polarizer, and a sugar solution, contained in a suitable glass tube, be introduced into the polariscope between the two nicol prisms, the polarized light will again pass through the analyzer; and the latter must be rotated to the right or the left through some degrees before the light is again shut off by total reflection. which so affect light are said to be optically active or to possess rotatory power, and the name circular polarization is given to the phenomenon, owing to the fact that the optically active substance appears to give a twist to the light ray. Without further discussion of the theory of such polarization, it is to be noted that those bodies which require the analyzer be turned toward the right in order, with the least rotation, to reach the position of total darkness, are called dextro-rotatory; those which require a rotation to the left, laevo-It is further to be remarked that, if the length of the tube containing the solution be fixed, the degree of rotation required bears a definite relation to the concentration of the solution. reason, the polariscope is useful not only in ascertaining the presence of dextro-or laevo-rotatory substances, but also for determining the quantity in which they are present in a solution, or in a substance from which the solution has been prepared; inasmuch as most of the common solvents are not optically active.

The sugars and dextrins have the further property of forming with water solutions of densities definitely related to the proportion of the respective solids in the solutions; hence, the specific gravity of the solution, that is, its weight, as compared with that of an equal volume of water at the same temperature, indicates very closely the quantity of these solids in solution. Furthermore, the refractive index of sugar or dextrin solutions, that is, the power of these solutions to bend a ray of light entering or passing from them into the air, also bears a definite relation, at a fixed temperature, to the percentage strength of the solution. For these reasons, both the specific gravity and the refractive index of a sirup are physical properties useful for the purpose of the analyst.

Dextrose, d-glucose, grape sugar, starch sugar  $(C_6H_{12}O_6)$  occurs, but usually in association with other carbohydrates, especially levulose and cane sugar, dissolved in the juices of numerous plants, especially in those of the sweet fruits. Honey contains it in large quantity, and often in the form of white crystals. Dextrose is obtained also when cane sugar is acted upon by certain ferments and

acids; and, in like manner, from malt sugar; in the former case, associated with levulose; and it is the end-product obtained when starch is acted upon by dilute acids, mineral and oxalic. Dextrose is sweet, but not more than three-fifths as sweet as cane sugar. be crystallized, the process is much more difficult than in the case of cane sugar. The product is now, however, manufactured extensively. Dextrose shares with certain other substances the property of multirotation; that is, it does not exhibit the same optical activity immediately after it is dissolved, as it does after the solution has stood for some time. After twenty-four hours standing, its dextro-rotatory power is constant. When heated at a temperature of more than 200° C. (392° F) it turns to caramel or sugar color, a brownish black substance of bitter flavor and high coloring power. When a solution of dextrose is heated with Fehling's solution (an alkaline copper solution), it reduces the copper to the form of an insoluble red suboxid, whose quantity can be determined and bears a definite relation to that of the dextrose, providing proper precautions are used. sugar is highly fermentable and can be directly acted upon in solution by yeasts and molds to form carbonic acid and alcohol. food for man, it has high nutrient value, being capable of digestion without previous chemical change. One gram of dextrose gives up when burned 3742.6 calories of energy; that is, heat enough to warm 3742.6 times its weight of water from a temperature of 4° C up to 5° C. It is changed in the animal body, uniting with other materials to form a variety of substances, of which glycogen, the sweet principle of the liver, is an important member. In the course of certain diseases, it fails either to be thus combined or to be fully oxidized to carbonic acid and water as the tissues waste, and therefore is excreted in the urine, producing the symptom, diabetes mellitus.

Levulose, d-fructose, fruit-sugar,  $(C_6H_{12}O_6)$  occurs with dextrose in plant juices and in honey. It too is formed when cane sugar is acted upon by weak acids and by certain ferments. It is also made by the action of acids upon inulin, the carbohydrate characteristic of the artichoke. Levulose is not a very stable body and is therefore difficult to prepare in a pure state.

It crystallizes less readily than dextrose. It is sold, in a nearly pure state, under the name of "diabetin." Levulose dissolves in water to form a solution sweeter than that of cane sugar. It decomposes readily on heating, even at a temperature as low as that of boiling water. For this reason, it is impossible to determine exactly, by evaporation under ordinary conditions, the quantity of solids dissolved in a liquid, if levulose be one of the components. It differs in optical activity from dextrose in that it has a left hand rotation, differing also in degree from that of dextrose, and also in the further particular, that its optical activity decreases rapidly with an increase in the

temperature of the solution under observation, whereas the optical activity of dextrose is little affected by temperature variations. Levulose like dextrose, exhibits multirotation. It too reduces Fehling's solution, and somewhat more vigorously than dextrose. Like the latter, it is capable of direct fermentation by some yeasts to form alcohol and carbonic acid, but its fermentation is slower than that of dextrose, and beer yeast is unable to set up alcoholic fermentation in a levulose solution. Levulose, also, is capable of direct absorption by the animal body without previous digestive change. It is undoubtedly assimilated in the same general manner as dextrose, but rarely appears in the urine. Its heat of combustion is about the same as that of dextrose.

When cane sugar solutions are carefully heated Invert Sugar: with oxalic acid, the sugar splits, combining with water in the process, into dextrose and levulose, in equal parts. Mineral acids also effect a like change, as well as certain unorganized ferments, which are therefore called invertases. A water solution containing about 25% of cane sugar, when mixed with one-twentieth of its volume of strong hydrochloric acid, and heated carefully so that in fifteen minutes its temperature is raised to 68° C (about 157° F.), is completely inverted. A like mixture inverts completely on standing for ten hours at 20°C (68°F.), and, in manufacturing practice, much smaller quantities of acid are used with complete effect, though a longer period is required for the change. The invert sugar solution thus produced is very sweet, and has often been used as an adulterant for honey. This sugar mixture is formed in considerable amounts whenever cane sugar solutions are heated in the presence of any acid, and, in small amount, even on prolonged heating of a pure sugar solution. It is also formed during most fermentations of cane-sugar, though, owing to the difference in the susceptibility of dextrose and levulose to alcoholic fermentation, the equality of these two sugars in the product from the splitting up of the cane sugar, is probably not long maintained; and the greater instability of levulose when heated, tends to produce a like result. In its behavior toward Fehling's solution, invert sugar resembles its two components. Owing to the unlike character of the optical activity of dextrose and levulose with respect to temperature variation, invert sugar, though strongly laevorotatory at ordinary temperatures, shows no activity whatever when heated with extreme care up to 87° C., as the opposite tendencies of the component sngars exactly neutralize one another at this temperathre; heated to a higher degree, the rotation becomes right-hand and, if the heat be high enough or sufficiently prolonged to cause some decomposition of levulose, remains right-hand even though the solution be cooled down to 87° C. This optical behavior of invert sugar is highly valuable for its quantitative determination.

Cane sugar, sucrose, saccharose, (C12H22O11) occurs, usually with dextrose and levulose, in a great variety of plants. Its principal sources for manufacturing purposes are the sugar cane, the sugar beet, and the maple. Although many attempts to prepare it artificially have been made, they have thus far failed. It occurs in commerce in beautiful white, rhombic crystals. Prepared in large crystals slowly formed in a concentrated solution of purified or refined sugar, it is known as "rock candy," and the residual solution as "rock candy syrup." When heated to 160°CC. (320° F.) it melts, forming upon cooling a yellow solid known as "barley sugar;" heated at slightly higher temperature it splits up into dextrose and levulosan, a substance formed when levulose is heated so as to drive off water; and at still higher heats, progressively yields glucosan or dextrosan and caramelan. When its solution is boiled with dilute acids, it is inverted, as previously stated, yielding equal parts of dextrose and levoluse. If a water solution of this sugar is boiled for a long time, it becomes acid and less viscid, owing to the formation of The action of sucrase or invertase, a soluble ferment invert sugar. extractible from yeast, results also in inversion. Cane sugar is not directly fermented by yeast to form alcohol and carbonic acid; not until it has been inverted, can the alcoholic fermentation proceed. Also, this sugar does not directly reduce Fehling's solution, although on long boiling some inversion occurs, whereupon a slight formation of the copper sub-oxid follows. Cane sugar is strongly dextrorotatory, but, upon inversion, its solutions yields laevo-rotatory liquids, that is, solutions of invert sugar. This optical behavior, before and after inversion, is valuable in determining the quantity of cane sugar in solution with the other sugars, the successive polariscopic readings being made at the same temperature and the cane sugar computed by the following formula:

Cane sugar (%) = 
$$\frac{(a-a^1)}{142.66-\frac{1}{2}} \frac{100}{T}$$
.

where a and  $a^1$  are the polariscope readings before and after inversion and T is the temperature at which these readings were made.

Cane sugar, like the monosaccharoses, has the power of acting in some relations like an acid. It acts rapidly and corrosively upon iron and steel, hence the use of copper and tin-plate vessels for confectioners' and preservers' purposes; also upon lead, taking this metal into solution, wherefore containers and faucets for holding and delivering sugary liquids, should not have leaden or lead-alloy parts in contact with the liquids; nor should lead-containing foil be used as a wrapper for confectionery, unless protected from contact therewith.

Cane sugar is regarded as, to some extent, directly digestible. Nevertheless, it is partially inverted in the stomach by the acid secretion and also very promptly upon entry into the small intestine. If injected into the bowel below the pancreatic duct, it leaves the body unchanged. From recent physiological experiments with man and domestic animals, it appears to have especial value as a prompt restorer of muscular vigor, and has of late been largely used by athletes and by soldiers on the march, and also fed in molasses to dray horses.

Malt contains two closely related disac-Malt sugar, maltrose. charose sugars, maltose  $(C_{12}H_{22}O_{11}+H_2O)$  and isomaltose  $(C_{12}H_{22}O_{11})$ . These sugars have many properties in common; for example, both are sweet, readily soluble in water, capable of reducing Fehling's solution but in different proportion to a unit weight of the respective sugars, and highly dextro-rotatory though in somewhat different Maltose has, when just dissolved, a much lower optical activity than some hours after the solution has been made. be crystallized in fine needles, but isomaltose has not thus far been The latter is far less readily fermentable than maltose, and is, according to Baer, not all fermented by "bottom" or ordinary beer yeast. Maltose itself does not undergo alcoholic fermentation by the yeast species known as Saccharomyces apiculatus and S. Maxianus. These differences in behavior are useful in separating these sugars, as is the great difference in certain of their compounds known as osazones, which need not be described for the purposes of this publication.

Both these sugars are produced in the progressive changes which starch undergoes in germinating barley under the influence of its soluble ferment, diastase. A like series of changes is produced by dilute mineral acids, such as sulphuric and hydrochloric, and also by oxalic acid at boiling heat. The starch grains burst, and their contents form what is known as "soluble starch," because of its solubility in water. Like raw starch, it is colored an intense blue by iodin. In turn it is changed to erythro-dextrin, giving a wine-red color with iodin, and progressively into a series of dextrins called achro-dextrins, because they give no characteristic color with iodin. According to Lintner and Dull, each step in this progressive dextrinization is accompanied by a splitting off of isomaltose, which is also yielded upon the breaking down of the last achro-dextrin; and the isomaltose, in turn, is then converted into maltose. Here the malting practically stops; whereas, on rather prolonged treatment with acid, maltose is split up to form with water the simple sugar, dextrose.

The digestion of starch exhibits a progressive series of decomposition products much the same as that shown in the germination and acid hydrolysis processes. The saliva, by action of the pytalin or salivary amylase it contains, carries the processes through to the maltose stage, providing the conditions allows it to act long enough upon the starch. The action is arrested by weak acid, such as that of the gastric juice; but, owing to the time required in the stomach for the complete mixing of the gastric juice with the food received from the mouth, salivary digestion probably continues for sometime after the arrival of the food in the cardiac, or heart end of the The saliva also contains some maltase or glucase, by which a small part of the maltose is transformed into dextrose. The salivary action of raw starch is very slow, but in the case of cooked starch the changes are set up instantly upon contact with saliva. the partially digested food is passed on from the stomach to the small intestine, it is mixed with several digestive secretions which there enter the alimentary canal. One of these, the pancreatic juice, secreted by the pancreas, or sweet bread, contains a number of digestive ferments, each having a specific function; of these ferments, we are particularly interested in sucrase, which inverts cane sugar, an amylase more vigorous than the ptyalin of the saliva in converting starch to dextrin and later to maltase, and finally a maltase or glucase by which maltose is in part hydrolyzed to dextrose.  $C_{12}H_{22}O_{11}$ ,  $H_2O$ , has a heat of combustion of 3721.8; isomaltose (?), of 3949.3 calories.

Dextrin, achro-dextrin: The occurrence and manufacture of these bodies may have already been sufficiently described, though it may be here noted that starch exposed to roasting temperature in converted to dextrin, which forms the glossy coat of bread crust. to be noted that they are white, non-crystalline bodies of rather gummy character, which dissolve in water forming very viscid solutions, but are thrown out of solution when enough alcohol is added to make the liquid moderately alcoholic (about 30%), whereas the sugars are soluble in 80% alcohol, though not in absolute alcohol. The dextrins are all highly dextro-rotatory, though they differ from one another in their degree of optical activity and in their alcohol solubility. They are devoid of any marked sweetness of flavor; have little, if any, power of reducing copper solutions; and are not directly fermentable by yeast. As already indicated, they require a more extensive change by digestive processes to fit than for assimilation than do the sugars.

### MAPLE SIRUPS

Maple sirups are made, directly or indirectly, from the sweet sap of various species of the maple. It might with fitness be called the American sirup, since it was crudely made by the Indians probably long before the first European settler came to the Western continent.

Today its manufacture is carried on in many States of the Union and also in Canada. Vermont, New York, Ohio, Michigan, Pennsylvania and New Hampshire are its principal American sources. According to Hills², no maple sugar is made south of 35° latitude, or west of 95° longitude.

Owing to the fact that this sirup is produced in Pennsylvania its production deserves somewhat extended consideration.

## THE SUGAR OF THE MAPLE AND ITS ORIGIN

The sugar of maple sap is saccharose, commonly called "cane sugar," the same, in other words, as the natural sweet substance of the sugar-beet, and the principal sugar in the juice of the sugarcane.

Though the tree is not in a condition of active growth at the time when the sap flow begins which is diverted to the sugar maker's use, and the substances dissolved in the sap are, except as they may be taken up directly from the soil, derived from reserve food supplies stored in the wood, during the preceding growing season, to meet the tree's needs early in the spring, the sugar itself is not found as such in the wood of trunk, root, or branch, nor in the bark, except it may be in traces.

Investigators had, long ago, observed the occurrence of starch in the winter wood and especially the root wood of the maple, and its disappearance upon the spring-time development of the buds and leaves, Storer<sup>3</sup>, in various studies of the source of the maple sugar, described the occurrence and arrangement of the starch in various parts of the woody tissues. With these findings, Spaulding and White, investigating the matter more widely by michrochemical methods under the direction of Jones<sup>4</sup>, were in close agreement. They found that the starch (and associated sugars) occurs chiefly in the cells and fibers of the sap wood; that it is most abundant in the medullary rays; and that, in general, it appears to be deposited in the outer cells first and then progressively inward.

As to the quantity of starch present in the wood, few determinations have been made. Storer<sup>5</sup>, by treatment with malt extract, obtained from various tissues of a sugar maple felled in May, reducing materials equivalent to the following percentages of starch in the water-

<sup>1.</sup> Small figures in the body of the page refer to reference lists.

free wood: Inner wood, 1.94%; outer wood, 2.43%; bark, 5.97%. No attempt was made in these determinations to separate the sugars before proceeding to the estimation of the starch. Any sugars present are therefore represented in these figures by their equivalent Elsewhere3, this author reports that he obtained from the wood of a sugar maple root by aid of malt extract, the equivalent of 5 to 7 per cent. of starch; in this case, the wood yielded to boiling water matters capable, without previous treatment with malt extract, of reducing Fehling's solution, and therefore assumed to be dextrose or invert sugar. The quantity was equivalent to 4.12% of dextrose. Storer judged the quantity of starch to be insufficient, however, to account for the amounts of sugar developed and the physiological work performed, observed that mannan also is present in the winter wood, but not in that of trees felled after the sap flow, and suggests that this constitutent also might serve as a source of cane sugar, which has been found in the seeds of coniferous trees, whose seeds contain mannan as a prominent constituent.

Combining the data given by Jones and Storer, it would appear, however, that the starch found by Storer is probably equivalent at least to the cane sugar formed in the tree during the season of sap flow, as indicated by the quantity of sap in the tree and the richness in sugar of that portion of the sap which flowed from the tap.

#### SAP FLOW

The Rock Maple or Sugar Maple (Acer saccharinum, Wang) is the principal source of maple sugar and sirup, but sugar-containing sap is also obtained from the Black Maple or Black Sugar Maple (A. saccharinum, Var. nigrum, Torrey and Gray; A. nigrum, Michx.), the White, Silver, Soft, or River Maple (A. dasycarpum, Ehrh.) the Red, Swamp, or Water Maple (A. rubrum, Linn.), the Oregon or Cabinet Maple (A. macrophyllum,) the Vine Maple (A. circinatum, Pursh.), the Striped Maple (A. Pennsylvanicum, Linn.), the Norway Maple (A. platanoides, Linn.), and also from the Ash-leaved Maple or Box-Elder (Negundo aceroides, Moench.) a species closely related to the maple; and most of these species are used as sources of commercial sirup and sugar.

All these species possess the property of copious bleeding from wounds during the later Fall and early Spring months, and sometimes even on sunny days in mid-winter. The sap flows most freely from the young wood, relatively little coming from the heart wood. The flow is most abundant near the base of the trunk. The flow occurs chiefly in day-time and is most abundant when the ground is covered with snow, the earth is moist, the days warm and the nights cold. Trees with a relatively large leafage and sunny exposure during the preceding summer, yield the most sugar.

The conditions and causes of this very special kind of sap flow have been extensively studied by Clark<sup>7</sup>, Schroeder<sup>8</sup>, Wood<sup>9</sup>, and the writer<sup>10</sup>, but the most searching and extensive investigations upon this subject are those recently made by Jones, Edson and Morse<sup>2</sup>, of the Vermont Agricultural Experiment Station. This phase of the subject can not be discussed in this connection. It appears, however, that the flow is probably the result of an intermittent internal pressure due to the expulsion of the sap from the interior of the living cells by the action of their protoplasm or jelly-like contents, and that this cell activity is a normal process and is more or less stimulated by the alternating atmospheric temperatures peculiar to the seasons of flow.

# QUANTITY AND COMPOSITION OF MAPLE SAP

The quantity of sap yielded by a single tree varies, of course, with its size and surroundings. The writer obtained from thirteen fair-sized trees in April, 1885, an average total flow, for the season, of 62.3 pounds, or about  $6\frac{1}{5}$  gallons per tree, though individual trees varied from about 9 pounds up to 171.7 pounds. In more favorable seasons, the flow would probably have greatly exceeded these quantities.

The white and red maples gave fully as much sap, in proportion to their size, as the rock maples.

Jones and his associates reached the same conclusion, but question the endurance of so great yields.

The average sugar richness of the sap of various trees is quite different, ranging, in the writer's experience, from 2.33 to 5.01% in a season of moderate richness. Jones reports a range of 1.33 to 7.149% for single days and single trees in 1900. Sap from the same tree varies in sugar richness during the season, but with no apparent regularity; and even at different hours of the same day, or at the same hour on different sides of the tree, shows differences in composition. Taps at different elevations on the same tree, not only yield much less sap from the upper than from the lower taps, but also a much richer sap, the flow from the upper taps often having an almost sirupy consistence.

The total sugar obtainable in a single season from a tree of size large enough for use, ranges from \( \frac{1}{3} \) to as high as 40 pounds; but the average is from two to five pounds, according to the season. The quantity of sap required for each pound of sugar made, is about 16 quarts, though for some seasons and some trees, the volume of sap needful runs as low as 5 quarts.

The juice from most sugar-containing plants usually contains, associated with the cane-sugar, relatively large amounts of dextrose and levulose. The juice of the sugar-beet is free from these sugars,

but, as appears in another part of this bulletin, the juice of the sugar-cane contains them in considerable quantities, while in the juice of the sorghum they are relatively so abundant as to make difficult the manufacture of cane-sugar from this source. In the maple sap, these reducing sugars are often absent, in the case of early run saps; but, as the time of bud development arrives, these sugars appear in the sap in small amounts. The writer found in eight saps taken near the last of April an average of 0.256% of reducing sugars, though excluding three "buddy" saps in which the quantities were exceptionally high, the average was reduced to .057%.

Starch was sought by the writer in a large number of saps without its discovery in any instance.

The saps do, however, all contain nitrogenous, acid, and mineral constituents. The writer found albuminoids (calculated from the total combined nitrogen present) in amounts ranging from .0069 to .0469%, the maximum quantity appearing in the late runs.

All the fresh saps have a fairly acid reaction, ranging in fresh juices, from .0005 to .005%, stated as malic acid. The sap of the maple is characterized by containing in quantities differing with the soil upon which the tree stands, an acid malate of lime. when the sap is concentrated, becomes insoluble and is deposited in grayish crystals to which the sugar makers have given the name "sand" and "niter." Some orchards yield sap containing such abundance of this acid compound, that it encrusts the evaporating pans and interferes seriously with the evaporating process, and later, as the sirup cools, separates forming a dirty looking gray, gritty deposit at the bottom of the container.\* The peculiar flavor of maple sirup and sugar is not readily perceptible in the sap, concentration being necessary before the flavor becomes distinct. The nature of the distinctive flavoring principle has not been satisfactorily determined. It can, however, be largely separated from the sirup by agitation with ether, in which the flavoring substance or substances are more soluble than in water.

The character of the mineral matters will be discussed when the composition of the sirnps is considered.

*Warren 13 states that, with care, 16-25 lbs, of this crude malate can be obta	bori	in o ci	næle
season. This composition of the malate is given by him as:	meu	m a si	ngie
Ferrie oxid	Per	cent.	
Ferrie oxid,	-	.39	
Calcium oxid,	_	17.16	
Magnesium oxid,		.03	
r nosphore acid,	_	.05	
Siliea,		7 74	

100.00

## MANUFACTURE OF MAPLE SIRUP

In early days, the making of maple sirup and sugar was carried on in a very crude way. The trees were tapped by cutting with an ax an obliquely upward gash, into which a chip was fitted to serve as a spout. The sap was collected in roughly hewn, wooden troughs and carried in pails to the nearby iron kettle set on or swung from a tripod. The boiling process was continued, with little or no purification of the juice, until the contents of the kettle were brought to the desired consistence. The rough method of tapping the trees was, however, very destructive to their vitality and the flow and color of the sirup were not superior.

The processes of today, though simpler than is the case with the manufacture of any other true sirup, show a very distinct advance over the earlier methods just described. The trees are tapped with a three-eighth inch bit and deeply enough to penetrate the sap-wood. A galvanized metal spout is driven into the tap-hole. From the spout is suspended a heavily tinned pail with a cover to exclude rain water, and prevent the access of birds, squirrels and other animals. The sap is gathered daily, gathering tanks of tinned metal placed upon wagons or sleds being used for the purpose, and from these gathering tanks the sap is transferred to the metal storage tanks of the sugar house.

In the present day practice, it is the aim to avoid storage of the sap, because of its liability to bacterial fermentation. In the latter part of the season, as the weather grows warmer, sap often assumes a green or greenish brown color, or becomes milky, or exhibits long strings of water-white glairy substance insoluble in alcohol, all of which yield dark-colored, off-flavored sirup and sugar. Edson<sup>14</sup> finds all these defects to be due to various bacteria, and recommends repeated cleansing of sap pails, the use of covered pails, and the avoidance of storage. In Ohio, where the season is rather long, the tap-holes are often bored out with a slightly larger bit, late in the season, to remove bacterial growths that have accumulated in the tap-hole.

It happens, not infrequently, that, during cold nights, the afternoon flow of sap freezes in the pails. The freezing is rarely complete; the result being that most of the sugar is concentrated in the unfrozen portion of the sap. Many sugar makers reject the ice as worthless, but Jones<sup>15</sup> has shown that, on the average of five observations, the ice contained over one-fourth of the original sugar, an amount probably sufficient to repay the cost of evaporation.

As already stated, the apparatus and process of evaporation have been markedly improved in recent years. From ordinary kettles, an advance was made to shallow sheet-iron pans resting on a brick arch, provided with flue and draft regulators. Today, long iron furnaces with improved draft regulation are used, upon which are

mounted, with a gentle inclination from rear to front, tinned pans of about six inches depth, provided with partitions set at right angles to the long axis of the pan, reaching but part way across the bottom, and alternately joined to the respective long sides of the pan, so that the sap, which is automatically delivered at the rear of the evaporator, may flow slowly from side to side as it moves forward. The stream is kept at a depth of about  $1\frac{1}{2}$  inches at the upper and 1 inch at the lower end of the pan.

As the sap is heated, the albumen is coagulated and rises with other impurities, so that they may be skimmed off. Milk is sometimes added in small quantity to facilitate the purification, but is undesirable, because it introduces foreign matters that unfavorably affect the excellence of flavor of the finished product.

The boiling is continued until the sirup has reached a concentration such that the boiling sirup has a temperature of 219° Fahr., corresponding to 11 pounds weight per gallon of sirup.<sup>16</sup>

If "niter" appears in the sirup, it may be removed by filtering the hot sirup through a flannel filter. If the lower sections of the pan become coated with the niter, it may be removed readily by reversing the position of the ends of the pans, so that the coated sections are exposed to fresh sap, by whose action the niter is redissolved.<sup>17</sup>

Concerning the effects of filtration, Jones notes that filtration through sand does not lessen the flavor intensity of the sirup, but that by filtering it through charcoal, not only is most of the color removed, but, as well, most of the distinctive flavor to which the sirup owes its high value as compared with simple sirups prepared from ordinary refined cane or beet sugar.

In many instances, the sirup, instead of being held as such, is further concentrated until it "sugars off." It is not germane to the subject of this bulletin to describe this process in detail. It should, however, be noted, since much commercial maple sirup is made by "melting," that is, dissolving maple sugar in water, that in the making of the sugar no molasses is separated and no chemical addition is made. In other words, all the constituents of the sirup, other than water, are retained unchanged, except by heat; so that the product is what is known technically in sugar manufacture as a "concrete." Sirups intended for sale as such, rather than for subsequent sugaring off, are usually clarified more carefully than where sugar is the end product.

#### GENERAL QUALITIES OF MAPLE SIRUP

As the process of manufacture above described, has doubtless made clear, maple sirup is maple sap, less water removed by evaporation and albuminoids with minute quantities of associated materials removed by skimming, and in some cases, less malate of lime removed by sedimentation or filtration; plus such conversion products as the original materials may yield under the influence of heat Or, if the sirup be made from maple sugar during evaporation. (concrete), it is simply maple sugar plus water.

Pure sirups may, before they reach the consumer, deteriorate, however, because of the attack of yeasts or molds, which convert the cane sugar to invert sugar or even completely destroy part of the sugars, and impart a musty, undesirable quality to the flavor. Sirups of too low body or concentration are especially subject to such attacks. Wherever possible, the sirup should be delivered hot into the containers, previously sterilized by heat, in which the sirup is to be delivered to the consumer.

As previously stated, maple sirups owe their value chiefly to the distinctive flavor, in adition to that of mere sweetness, which they possess. These sirups exhibit, however, quite a range of quality as respects this distinctive flavor, the corresponding odor, and color.

Jones<sup>18</sup> thus describes these three qualities:

"Aroma.—The odor of maple products is at once pleasing and characteristic. That it is due to some partially voltile principle, the odor of boiling sap or syrup makes plain. This aroma is at its best when the goods are fresh. It is apt to be bound up' in old and dry samples as to be apparently lacking." "Flavor.—The flavor or taste of maple goods may serve in a measure to indicate their purity. Inasmuch as many pure maple syrups, particularly those produced in certain sections of the country, have what might be termed a 'molasses taste,' it is best not to form hasty opinions from this sense alone, especially if the origin, age, and conditions of manufacture are not known. The flavor, taken into consideration with color, is a good criterion as to quality of maple products." "The general characteristic of maple syrups from different sections of the sugar producing areas that have come under the writer's notice may be thus briefly described. Remarks refer to average quality goods:

"Pennsylvania: Sweet and flat (often like molasses); maple flavor.
"Ohio: Mild, delicate almost to flatness: maple flavor.
"Vermont: Mild, delicate; good maple flavor.
"New York: Strong; maple flavor.
"Canada: Good maple flavor. Bulk of product dark in color, with strong flavor, occasioned by demand.

"As far as marketable differences go, there is but small choice between Vermont, New York and Canadian goods of equal grade."
"Color.—The color of maple syrup and sugar is no indication of purity. Pure goods may be almost snow white, or they may be nearly jet black or any intermediate shade of brown. The color of pure goods is largely dependent on the methods used in handling and boiling the sap. The careless operator, even with the best utensils, will produce a poor grade, highly colored syrup or sugar. On the other hand, his cleanly and careful neighbor will make a first grade material."

## GENERAL COMPOSITION OF MAPLE SIRUP AND MAPLE SUGAR

From what has been earlier stated, it is clear that the degrees of concentration and purification of these products vary with the aims and skill of the manufacturer, and, in a measure, also with the character of the sap which serves as his raw material. A general idea of the composition of these products may be obtained from careful analyses of authentic sirups collected by the writer in Vermont<sup>19</sup> and of authentic sirups and sugars collected by Hortvet<sup>20</sup> from New Hampshire, Vermont, Conecticut, New York, Pennsylvania,

Wisconsin, and Minnesota, although no true average for the manufacture as a whole can be secured from the data afforded by the thirty-two sirups and nineteen sugars represented in these averages. The latter are as follows:

	Maple syrups	Maple sugars.
Moisture, Cane sugar, Invert sugar, Albuminoids, Ash, Undetermined,	% 33.87 59.88 2.75 .196 .64 2.664	% 8.85 84.77 2.23 .495 .91 2.745
	100.000	100,000

It is of prime importance to the consumer that he obtain the genuine maple flavor in as concentrated form as practicable; or, at least, that the product he buys shall be as well standardized as conditions allow in respect to this principal quality of the maple products. The normal variations with the quality of the raw materials are sufficiently wide to meet the divergent tastes of consumers and the corresponding demands for delicate, or for relatively rank flavors, without a further modification of flavor intensity by a graduation of. the concentration of the sirup.

The Secretary of Agriculture21 of the United States has proclaimed, under authority of Congress, the following standards for these products:

"Maple Sirup is sirup made by the evaporation of maple sap or by the solution of maple concrete, and contains not more than thirty-two (32) per cent. of water, and not less than forty-five hundredths (0.45) per cent. of maple sirup ash."

"Maple Sugar is the solid product resulting from the evaporation of maple sap,

and contains, in the water-free substance, not less than sixty-five one-hundredths (0.65) per cent. of maple sugar ash,

Pennsylvania has no statutory, or regulative standard for maple products, but many of the States have, either by law or by duly authorized regulations, adopted the standards proclaimed by the Secretary of Agriculture.\*

<sup>\*</sup>The following standards for maple products were promulgated, March 22, 1911, by the Dominion of Canada, 22 by order in Council, under Sec. 26, Adulteration Act.

"Maple syrup is syrup, made by evaporation of maple sap, or by the solution of maple concrete in water; and contains not more than thirty-five (35) per cent. of water. The total ash, reckoned as a percentage on the dry matter of the syrup, shall not be less than 0.5 (five-tenths of one per cent.) The malic acid determined in prescribed manner, shall not be less than 0.4 (four-tenths of one per cent), reckoned as a percentage on dry solids. The lead subacetate number, determined as prescribed, shall not be less than 2.2 (two and two-tenths)."

For reasons later stated in the body of this bulletin, the Canadian officials propose to reduce the malic acid to 0.3 per cent. and the lead acetate to 1.7, and also to fix a minimum limit for insoluble ash at 0.12 per cent. of the dry solids.

The National standard for maple sirup corresponds to a density of 1.3384. The general practice of maple sirup makers is to accept 11 pounds to the gallon, corresponding to a density of 1.325, as the desirable degree of concentration. Thus, speaking of the practice in Vermont, Cooke and Hills<sup>16</sup>, writing in 1891, say:

"Most sugar makers boil down syrup until it weighs 11 pounds to the gallon and then let it cool. The particular point in making syrup is to get it as thick as possible without having it granulate on standing. The ordinary rule of the maker is to make syrup that shall weigh 11 pounds to the gallon, and we have found by experiment that this custom is exactly right and that the temperature corresponding to this weight is 219°. That is, if the syrup is taken off from the fire as soon as it shows a temperature of 219° it will weigh exactly 11 pounds to the gallon and will not grain on standing. That is, however, the extreme limit; if the temperature is allowed to get a single degree higher, the syrup will granulate. In practice, not much of the syrup that is on the market is quite up to this point, most of it being taken off just before it reaches 219°, and large quantities were on the market last spring (1891) which boiled at 216° or even less."

Hubbard<sup>17</sup> too, in writing in 1906, said, "When the sirup reaches a temperature of 219° F., or a weight of 11 pounds to the gallon, a deposit of malate of lime or 'niter,' will be observed. This temperature and weight are proper for good sirup."

In reference to the indicative temperature above mentioned, it should be remarked that the boiling points of liquids decrease with the altitude above sea-level of the place of observation. Sirup makers living at altitudes over 500 feet above sea-level should determine for their own locality the proper final boiling point corresponding to the standard density for maple sirup.

Another point relative to the concentration of the sirup deserves notice, viz: That, the denser the sirup, the less its tendency to deterioration by ferments,—another reason on the consumer's part for desiring a concentrated, rather than a watery sirup.

The sugars shown in the average analyses stated on an earlier page, exhibit more invert sugar than the traces present in fresh sap would lead us to expect. It is known that on long boiling, the cane-sugar of maple sap is inverted in considerable quantities; but since the concentration in a modern evaporator takes but a short time, sirups of standard density have been made from clean, fresh sap so as to show little more than 0.5% of invert sugar in the finished product. Slow boiling in sugaring off also increases the proportion of inversion, which would be manifest in sirups made from the resulting sugars. High invert sugar is chiefly attributable to fer-

mentation of the sirup, or, in less degree, to souring of the sap by careless gathering and storage.

It is worthy of note respecting the sugars, that Storer<sup>26</sup> was unable to isolate mannose from maple sugar, although mannan was found in maple trees.

The analyses show, in addition to moisture and sugars, considerable quantities of other materials, including ash and "albuminoids." Concerning the ash, full discussion will be given in a later paragraph. The quantity of albuminoids is determined by calculation from the total nitrogen yielded by the sirup or sugar. As a matter of fact, it is not probable that the nitrogen is chiefly present in albuminous compounds, since true albumen would be coagulated like the white of an egg by the heat of the evaporation and largely removed in the skimming and filtration. The nitrogen is probably present more largely in less complex, soluble compounds that are not coagulated by heat.

Among the undetermined matters, are acids, including malic acid, and the brown or reddish brown materials of the sirup and sugar.

### ADULTERATION OF MAPLE SIRUP

Owing to the relatively high price of maple as compared with ordinary cane and beet sugars and with glucose, the practice of mixing maple sirup with sugar solutions prepared either from ordinary refined sugar or from crude brown sugar, has been very general, as well as that of mixing with glucose. Admixtures of the latter kind were readily detected not only by study of the optical behavior of the mixture, but also by the peculiarly thick or viscous consistence of the mixture. A representative of certain manufacturers of such adulterated products stated to the writer several years before the passage of the National Food and Drugs Act, that fully 95% of the so-called "maple sirups" on the market were either thus compounded or wholly spurious, and urged the great excess of demand over supply of maple products as a satisfactory reason for tolerance of this wholesale fraud—for all these products were sold under the name of the genuine article.

Among the imitations upon the market have been found ordinary sugar sirups flavored with extracts from hickory or maple wood or bark, or with distillates prepared therefrom. Recently a flavoring preparation, known as "Mapleine," has been put upon the market a very little of which is said to impart a maple flavor to a relatively large quantity of sugar sirup. Another imitation quite satisfactory to many palates is made by boiling sugar sirup with corn cobs, from which a pleasant flavor is extracted.

There is no good reason why such imitations should not be made for home consumption, if they are agreeable to the consumer's taste, but there is no sound excuse for vending them under deceptive names.

# DETECTION OF ADULTERATION OF MAPLE SIRUPS.

Owing to the similarity of the principal constituents of maple sirup and those of the common compounds and imitations thereof, a study of the sugars present offers no clue to the fact of adulteration, except where glucose mixtures are concerned; and, in the absence of conclusive means of distinguishing and estimating the characteristic maple flavor, recourse must be had to other differences, not in themselves directly affecting the values of the respective commodities, as proofs of the fact and character of the adulteration.

Maple Sirup Ash: The quantity of the maple sirup ash has long been recognized as a means of distinguishing the genuine sirup from the sirups made from refined cane sugar. Jones<sup>27</sup> and Hortvet<sup>28</sup> first studied extensively the properties and detailed compositions of the maple sirup and sugar ash as means of differentiating maple sirup from the adulterated and spurious sirups on the market.

Bryan<sup>29</sup> has compiled the results of these investigations together with those of about 60 authentic samples examined in the Bureau of Chemistry as follows:

CALCULATED ON ORIGINAL SUBSTANCE

	2	Iaple Sugar		7	Iaple Syrup	
	Minimum	Maximum	Average	Minimum	Maximum	Average
Water, (Per cent.),	3.05	11.00		Not	more than	32.00
Ash: Total, (Per cent.) Soluble. (Per cent.), Insoluble, (Per cent.),-	0.64 0.33 0.20	1.32 0.67 0.87	0.91 0.46 0.46	0.46 0.21 0.14	1.01 0.63 0.56	0.60 0.38 0.23
Alkalinity of ash:* Soluble,Insoluble,	0.40 0.55	0.95 1.72	0.63 0.94	0.26 0.31	0.68 0.94	0.50 0.54
Ratio of insoluble to sol- uble ash,	0.5	2.2	1.00	0.6	3.2	1.7

<sup>\*</sup>CC.N/10 acid required to neutralize ash from 1 gram of syrup or sugar.

Since the foregoing summary was written, the Chief Analyst<sup>22</sup> of the Inland Revenue Department of Canada, has published the result of analyses of 115 samples of maple syrup submitted by manufacturers with a declaration of purity and description of the methods of manufacture, many of which include the latest improvements in clarification and filtration. These gave a total ash: maximum 1.38; minimum, 0.69; average, 0.89 per cent.; and of insoluble ash: maximum, 1.01; minimum, 0.23; average, 0.37 per cent. Of the total ash, only one sample showed less than 0.70%; of the insoluble ash, only two samples had less than 0.19%.

In the absence of moisture determinations in many of the samples included in the foregoing summary, it was impossible to express the data on the basis of the dry matter of the sirups, the only basis

affording means for strict comparison. The analyses made by Jones in 1905, representing 48 authentic sirups and 43 authentic sugars, have, however, been computed on the dry basis, as follows:

CALCULATED TO DRY SUBSTANCE.

	i	Maple Sugar			Maple Syrup	
	Minimum	Maximum	Average	Minimum	Maximum	Average
Ash:						
Total, (Per cent.),	0.71	1.47	1.01	0.77	1.32	0.99
Soluble, (Per cent.), Insoluble, (Per cent.),-		0.70	0.53 0.48	$0.45 \\ 0.25$	0.72 0.78	0.58 0.39
Alkalinity of ash:					00	0.0
Soluble,	0.44	0.89	0.68	0.46	1.02	0.79
Insoluble,	0.61	1.91	1.01	0.55	1.45	0.83
Ratio of insoluble to sol-						
uble ash,	0.5	2.2	1.1	0.7	2.6	1.7

Analyses by Jones and by the Inland Revenue Laboratory of authentic Canadian syrups and sugars show that they fall within the above mentioned limits.

Jones and Hortvet studied also the detailed composition of the ash of maple sirup and sugar. A summary of the results obtained by the former is as follows:

	In 100 Parts of the Ash				Ratio of			
	CaO	$ m K_2O$	808	CaO to (K2O) 100	CaO to (SO <sub>3</sub> ) 100	K <sub>2</sub> O to (SO <sub>3</sub> ) 100		
Minimum, Maximum, Average,	18.03 31.74 22.34	18.26 38.98 31.45	0.68 2.42 1.65	57 181 141	3.4 12.7 7.4	1.9 9.4 5.3		

The ratios obtained by Hortvet fall within these limits, except that his maximum for the ratio CaO:  $K_2Ox100$  is 17.3, and the ratios CaO: $K_2Ox100$  obtained by Julius Schroeder in the ash of saps from the Norway maple also come within the foregoing limits for this ratio, except in the ease of the sap taken from the root of the tree.

At this point we may briefly consider the question whether the clarification of sirups by settling or by filtration may not diminish the quantity of ash and alter its composition, inasmuch as the niter held in suspension in the original sirup or in the solution obtained by "melting" maple sugar, is removed by filtration.

E. N. Eaton recently published results bearing upon this question, and affirmed the need for caution in the condemnation of commercial clarified maple sirups when the limits of quantity or of ratio of components were not fully met.\* But Jones and Hortvet, in the studies earlier made, experimented upon the effects of filtration processes which they regarded as more severe than those of commercial practice, and concluded that, while the filtration processes undoubtedly wrought changes in these respects, they did not cause the sirups to fall below the established minima, which Jones expresses as,

"Total ash, not less than 0.50%. Insoluble ash, not less than 0.15%."

The foregoing figures may be compared with those obtained from brown sugars of various grades, the base of most of the substitute or compound sirups. (Bryan)<sup>33</sup>

	Minimum	Maximum	Average
Ash: Total, (Per cent.), Soluble, (Per cent.), Insoluble, (Per cent.),	0.50 0.46 0.06	4.33 2.74 1.59	1.70 1.29 .41
Alkalinity of ash, (Per Ce.): Soluble,	0.15 0.15	$0.76 \\ 2.34$	.39 .82
Ratio of insoluble to soluble ash,	1.7	16.7	6.2

For raw cane sugar, the filtered sirups prepared therefrom, and beet sugar, the corresponding data compiled by Bryan are as follows:

	Raw Cane Sugar Cane Sugar Syrups			range			
	Minimum	Maximum	Average	Minimum	Maximum	Average	Beet sugar ra
Ash: Total, (Per cent.), Soluble, (Per cent.), Insoluble, (Per cent.),	.32 .10 .18	.59 .41 .23	.46 .25 .21	.19 .11 .08	.26 .16 .10	.24 .14 .09	.33— .86 .31— .78 .02— .08
Alkalinity of ash. (Cc.): Soluble, Insoluble,	.18 .42	.36	.29 .47	.22	.24	.23	.38— .40 .02— .28
Ratio of insoluble to soluble ash,	0.5	2.3	1.27	1.4	1.6	1.5	9.8 -15.5

These data show that brown sugar contains more ash, far more soluble ash, and usually a relatively more alkaline insoluble ash, than maple products possess.

<sup>\*</sup>Eaton's analyses of syrups before and after settling show very marked difference at the points under consideration. Critical comparison of his results renders doubtful, however, the representative quality of his samples.

Jones found also in brown sugar ash: Lime, 4-21.6%; potash, 30.7-55.4%; sulphuric acid (SO<sub>3</sub>), 4.6-17.8%. The corresponding ratios were:

Lime: potash x 100,	257 to 949
Lime: sulphuric acid x 100.	97 to 117
Potash: sulphuric acid x 100,	11 to 58

In other words, the proportions of sulphuric acid and potash, as compared with lime, in the brown sugar ash are far greater than in the maple product ash.

The properties of raw cane-sugar ash fall, it is true, largely within the range of those for maple product ash; but this is not the case with filtered sirups prepared in the usual manner from the raw cane sugars, since the ash of such sirups is less in quantity, at all points, and of lower alkalinity than the maple sirup and maple sugar ashes. Beet sugar ash is distinguished by a low proportion of insoluble ash, and by a correspondingly low alkalinity for insoluble ash, and by a high ratio of the latter to the soluble ash.

Lead Compounds: All genuine maple products yield precipitates upon the addition of lead sub-acetate to their solutions in water. This is due to the fact that the acids, albuminoids and gummy matters present, unite with the lead to form insoluble compounds. Raw cane sugar and cane sirups exhibit a similar behavior, but sirups made from refined sugar give no precipitate by this test. more, the quantities of the precipitate obtained from unit weights of these several products are more or less characteristic. and Jones 27 have devised a method for the centrifugal separation of these precipitates and the measurement of their volumes. methods require a high degree of exactness in order that closely comparable results may be obtained, but serve well to distinguish between genuine maple sirup and such as are chiefly composed of refined sugar. Sy has made a simple modification, depending upon subsidence for a fixed time rather than upon centrifugal force for the uniform separation preparatory to measurement of the volume of the precipitate.

A more exact method has been worked out by Winton<sup>35</sup>, who adds to the diluted sirup or sugar solution an exactly measured quantity of a standard solution of basic lead acetate, the quantity being in excess of the amount required to combine with the various reacting substances present, carefully separating by filtration the insoluble lead compounds, and determining exactly the residual lead in the solution. By difference, the lead taken into combination is determined. The weight of lead combining with the reacting substances in 1 gram of the sugar or sirup is termed the "lead number" of the maple product under examination.

Winton found that 8 genuine maple sirups gave lead numbers of 1.20 to 1.77; and 7 maple sugars of 1.83 to 2.38, adulterated samples

of sirup gave from 0.022 to 0.92. Bryan<sup>26</sup> states the range in lead numbers from all determinations made up to 1908, upon authentic samples, is as follows:

McGill has devised a simple method in which the washed subacetate precipitate is dried, weighed, and the weight multiplied by the factor 22.22 (assuming 10% of water in maple sugar), to give the lead subacetate number for the maple sugar equivalent of the sirup used. The Winton lead number is 71% of the lead sub-acetate number obtained by McGill's method.

Of 456 manufacturers' samples of maple sirup submitted with a declaration of purity and description of the process of manufacture, 31 samples gave lead subacetate numbers below 2.00%; 29 of these lying between 1.50 and 2.00%, and two falling below 1.50%.

Of these manufacturers' samples, 47 examined by the Winton method gave lead numbers as follows: maximum 2.38; minimum, 1.05; average, 1.75.

McGill concludes that the improved methods of manufacture tend to decrease these lead numbers to figures at least as low as 1.50 for the lead subacetate number, and 1.10 for the Winton number.

Malie acid value: Advantage has been taken of the facts that the distinctive acid of maple products is malic acid and that, when, to a hot neutral solution of this acid, dissolved calcium chlorid and strong alcohol are added, calcium malate is precipitated as an insoluble salt, which may then be separated and weighed. Hortvet<sup>37</sup> adapted to this purpose the method devised by Leach and Lythgoe<sup>38</sup> for the estimation of malic acid in cider vinegar. method does not involve the weighing of the insoluble calcium salt, but its destruction by burning and the determination of the quantity of standard acid required to neutralize the residual lime or lime carbonate, yielded by a standard weight of the maple product examined. The precipitate actually contains compounds other than calcium malate, but the rather arbitrary method gives "malic acid values" which are, nevertheless, quite characteristic for maple products. Hortvet and also Jones applied this method to authentic samples and obtained results of somewhat divergent range:

# MALIC ACID VALUE

Maple syrup: Hortvet, Jones,	0.84 to 1.76, average 1.07 0.41 to 0.72, average 0.53
Maple sugar: Hortvet. Jones,	0.98 to 1.67, average 1.29 0.65 to 0.84, average 0.75

That is, for some reason not apparent in the descriptions of the work, Jones obtained values about one-half as great as those reported by Hortvet.

McGill<sup>22</sup> examined 452 maple sirups submitted as genuine by their makers, and found malic acid values ranging from 0:50 to 1.00 in 96% of the samples. He says, "It is safe to infer that samples giving less than 0.5 or more than 1.0 as malic acid values, are exceptional. But it must be borne in mind that individual samples, guaranteed as genuine, give as low a malic acid number as 0.30; while 2% of the present collection gave malic acid values below 0.4." This investigator thinks that simp from the soft maple has a lower malic acid value than that from the hard maple.

The method has value as confirmatory of the lead numbers, which represent practically the same constituents of the maple products. Hortvet obtained from brown sugars, malic acid values of 0.08 to 0.18; and Bryan notes an average of 0.35 for filtered sirups prepared from raw cane sngar, and of 0.08 for beet sngar. Jones observes that the malic acid value of a filtered maple sirup, whether made by direct evaporation or by the melting of maple sugar, is only about four fifths of the value exhibited immediately prior to filtration. Sy<sup>34</sup> has suggested two additional preliminary tests.

Foam test: On the observation that maple products foam more when shaken than do cane sugar sirups, the following test was based: In a narrow cylinder graduated to 0.1 cubic centimeters, mix 5 cc. of sirup and 10 cc. of water by vigorous shaking for one-half minute. Allow the cylinder to stand for ten minutes, and then measure the volume of the foam on top of the mixture.

According to Sy, maple sirups show from 3.0 to 6.0 cc.; average, 4.1 cc. of foam; while adulterated or mixed products give less than 3.0 cc.

foam; while adulterated or mixed products give less than 3.0 cc.

Color test: Noting that the color of maple products is due in part to caramel, but in part also to other bodies; that caramel is insoluble in amyl alcohol, the other coloring bodies soluble; that while the latter are soluble also in water, they are relatively less soluble in water containing free phosphoric acid, Sy devised the following test: In a narrow tube or cylinder, to 15 cc. of the sirup (or 15 grams of sugar made up with water to 15 cc. volume) add 3 cc. of pure amyl alcohol and 1 cc. of a 20 per cent. solution of phosphoric acid, shake thoroughly, let stand until the alcohol separates and then note the color of the amyl alcohol layer. According to Sy, pure maple products always give a decided brown color; cane sugar products colored with caramel, no color; mixtures, from a trace of color to a light brown, according to the quantity of maple present.

Aroma test: Jones<sup>40</sup> declares that, upon heating, maple products emit an aroma distinguished readily from those given off by all maple imitations. The hetter to set free the volatile aromatic principles, which appear to become "bound" in old goods, 20 to 40 cc. of water is mixed with 20 grams of the material to be tested, and then the vessel containing the mixture is gently heated to holling upon an asbestos plate. The mixture should be so diluted as to boil at 215° C.

For the application of this method, a wide range of experience with known

For the application of this method, a wide range of experience with known materials is manifestly required.

### EXAMINATION OF MARKET MAPLE SIRUPS

Of the official samples of sirup submitted for examination, 56 purported to be maple sirups, pure or compound. The labels of 31 samples indicated the goods to be compound; those of 25 represented the sirnps to be pure maple sirups.

In view of the misbranding prohibitious of the Food Act of May 13th, 1909, these samples were divided into two groups, the first of which purport to be pure; the second, compound.

### MAPLE SIRUPS PURPORTING TO BE PURE

#### Group I.

16.

3261

In the following tabulations are stated:

- a. The group number.h. The Special Agent's serial number.
- The name and address of the manufacturer or distributor, where they are stated on the label.
- d. The brand name of the sirup.

  e. Any added remarks concerning the purity of the goods, or the quantity contained in the package.
- The name and address of the vender from whom the sample was purchased The price paid for the sample. f.
- g.
- Appended to the brand name signifies that the label declares that a guaranty of conformity of the article to the requirements of the National Food and Drug Act of June 30, 1906, has heen filed with the Secretary of Agriculture by the manufacturer.
- Signifies that the container is made of tin-plate; unless otherwise indicated, the containers are of glass.

Group No.	Agent's No.	
1.	3243	Finley Acker Co., Philadelphia, Pa. "H. G." Extra Fancy Genuine Vermont Maple Syrup, Absolutely Pure. Finest Obtainable. Bought of Finley Acker Co., Philadelphia, Pa. Price, 35 e.
2.	3149	Austin, Nichols & Co., New York: Sunbeam Brand Maple Syrup.* Bought of Market, 81 S. Main St., Wilkes-Barre, Pa. Price, 30 e.
3.	3213	Austin, Nichols & Co., New York: Sunbeam Brand Maple Syrup.* Bought of Gohl & King, Williamsport, Pa. Price, 25 e.
4.	3247	Bay State Maple Syrup Co., Boston, Mass.: Mount Washington Maple Sap Syrup, Highest Quality. Absolutely Pure. Bought of Showell, Fryer & Co., Juniper and Market Sts., Philadelphia, Pa. Price, 25 c.
5.	3158	Curtiee Brothers Co., Roehester, N. Y. Blue Label Sap Maple Syrup. Guaranteed Absolutely Pure. Bought of Franklin Grocery Co., Laneaster, Pa. Price, 35 e.
6.	3228	The Wm. Edwards Co., Cleveland, O. Edwards Pure Maple Syrup.* Bought of Wm. Schultz, 2426 Peach St., Erie, Pa. Price, 40 e.
7.	3240	Gimbel Bros., Philadelphia, Pa. Distributors: Lenox Brand Sap Maple Syrup. Bought of Gimbel Bros., Philadelphia, Pa. Price, 35 e.
8.	3198	The Great Atlantic and Pacific Tea Co., Jersey City, N. J. Distributors: A. & P. Pure Maple Syrup*. Bought of A. & P. Tea Co., 614 Penn St., Reading, Pa. Price, 10 c.
9.	3260†	M. J. Hapgood, Peru, Vt. Pure Vermont Maple Syrup. Bought of Abraham & Bro., 693 Northampton St., Easton, Pa. Priee, 28 e.
10.	3206	Name and Address of manufacturer not given; Hazen's Vermont Syrup, Warranted: Hobbs Bros., 26 Centre St., Shenandoah, Pa. Metal screw eap marked "Hazen's Pure Vermont Maple Syrup." Price, 25 e.
11.	3160	Huntington Maple Syrup & Sugar Co., Huntington, Vt., and Providence, R. I. Gold Leaf Brand, Pure Vermont Syrup. Put up expressly for First Class Trade. Bought of D. P. Lentz, Lancaster, Pa. Price, —.
12.	3156	The F. N. Johnson Co., Bellefontaine. O.: Native Purity Pure Maple Syrup.* Bought of Jno. L. Binkley, Lancaster, Pa. Price, 40 c.
13.	3148	Francis H. Leggett & Co., New York. Distributors: Premier Pure Sap Maple Syrup. Bought of O. G. Coursen, Seranton, Pa. Price, 30 c.
14.	3214	New England Maple Syrup Co., Fairfax and St. Johnsbury, Vt., and Boston: New England Vermont Maple Sap Syrup, Choicest Quality. Absolutely Pure. Bought of C. W. Hicks, Market St., Williamsport, Pa. Price, 45 e.
15.	3203	Rigney & Co., Rutland, Vt., and Brooklyn, N. Y.: Colonial Brand Pure Vermont Maple Syrup. "We guarantee this syrup to be absolutely free from gluco.", preservatives, or other adulterants." Bought of E. E. Brobst, Cor. Jordan and 7th Sts., Shenandoah, Pa. Price, —.

Rigney & Co., Brooklyn, N. Y. Colonial Brand Pure Maple Sap Syrup.\* Bought of Floyd Bush, 45 Fourth St., Easton, Pa. Price, 35 c.

0.	No	
Group No.	gent's	
ron	gen	
9	Y	
17.	3147	S. B. Rogers, Elmira, N. Y. Pure Maple Syrup. Bought of E. G. Coursen, Scranton, Pa. Price, 50 c.
18.	3162	R. C. Seldomridge, Laneaster, Pa. Sap Maple Syrup, Absolutely Pure. Bought of R. C. Seldomridge, Laneaster, Pa. Price, 30 c.
19.	3279	Sprague, Warner & Co., Chicago, Ill. Distributors: Ferndell Brand Maple Syrup. (One Pint.) Bought of Gettysburg Department Store, Gettysburg, Pa. Price, 35 c.
20.	3202	F. G. Strohmeyer & Co., New York. Pure Maple Syrup, Delicious and Wholesome. Bought of S. Davies, Shenandoah, Pa. Price, 25 c.
21.	3229	The Towlc Maple Product Co., St. Johnsbury, Vt., and St. Paul, Minn. Towle's Vermont Mald Brand Sap Maple Syrup.* (Log Cabin Trade Mark) (One Pint Full Measure). Bought of Kaufiman, Pittsburg, Pa. Price, 30 c.
22.	3230†	M. H. Tweed & Co. (M. H. Tweed, Harry F. Botsford), Pittsburg. Tweed's Maple Syrup, Absolutely Pure ("Guarantee: Maple Syrup made directly from the sap is much superior in flavor to that made from maple sugar. We guarantee this to be absolutely pure sap syrup"). Made from the sap of the maple. Bought of Kauffman's, Pittsburg, Pa. Price, 40 c.
23.	3126	Welch Bros. Maple Co., Burlington, Vt., Vermont's Fincst Quality Pure Maple Sap Syrup.* Green Mountain Bay. Bought of Dives, Pomeroy & Stewart, Harrisburg, Pa. Price, —.
24.	3152	R. C. Williams & Co., New York, Distributors: The Famous Royal Scarlet Brand Genuine Sap Maple Syrup.* Bought of Mohican Co., Wilkes-Barre, Pa. Price, 45 c.
25.	3207	R. C. Williams & Co., Cor. Hudson and Thomas Sts., New York. Elite Maple Syrup. Bought of Hobbs Bros., Shenandoah, Pa. Price, 25 c.

### EXAMINATION OF THE SIRUPS

The maple sirups were examined by both physical and chemical tests, as follows:

Physical:	
1.	Refractive index at 28° C. by use of an Abbe refractometer with heating prism.
2.	Specific gravity at 20° C. by use of the Westphal balance.
3.	Polarization by use of a Schmidt-Haensch double-wedge polariscope, standardized by use of standard quartz plates: Readings were taken (a) Direct at 20° C.; (b) after inversion by the Clerget process, at 20° C.; and (c) after inversion at 87° C., and are expressed in degrees, Ventzke.
4.	Sy foam test.
5.	Sy color test.
Chemical:	
6.	Total solids, computed from the refractive indices by use of Geerlig's table. The selection of this basis of computation rather than the specific gravity, was determined by Bryan's studies of the accuracy of the method as applied to maple products.
7.	Cane-sugar (sucrose) by use of the Clergot formula; expressed for direct and invert readings taken at 20° C.:  (Direct reading—indirect reading, Ventzke degrees) X 100
	Sucrose =
8.	Reducing sugars (Invert sugars) by copper reduction (Munson and Walker's table).
9.	Total asb.
10.	Water-soluble ash (by difference).

11.	Water-insoluble ash.
12.	Alkalinity of water-soluble ash, per gram of sirup.
13.	Alkalinity of water-insoluble ash, per gram of sirup.
14.	Ratio, Water-soluble ash.  Water-insoluble ash.
15.	Sulphuric acid (SO <sub>3</sub> ) in total ash.
16.	Lead number (Winton).
17 18.	Malic acid value (Hortvet). Qualitative test for benzoic and salicylic acids, and for saccharin.

These determinations, except as noted below, were made according to the official methods, or, in case where no method has been officially adopted, as described by Bryan.

GROUP I. "PURE" MAPLE SIRUPS
Physical Properties and General Composition.

Group Number.  Agent's number.  Agent's number.  Agent's number.  Agent's number.  Agent's 1,4573  3231  1,4557  3238  1,4564  3238  1,4568  3260  1,4538  3260  1,4538	 3£		T Olai	r Olalization ( )	.( ^)	-		(•;	ព្រះ			, asa , ats
	Refractive index C.	Specific gravity.	Before inversion at 20° C.	After inversion at .0 °02	After inversion at 87° C.	Sy foam test. Oc.	Moisture.	• Total solids (Geerlig	Sucrose (Clerget form	Invert sugar.	.dsA	Acids, gum, albur coloring matter, By difference.
	1,4573	1,328	+62.0	-18.92	+4.84	4.5	970	% 67.25	59.45	5.00	%	2.35
	1,4547	1,331	++ 28:75 27:75	19,25	25°8	0.0	33.83	66.15	52.69	6.22	89. 8	
	1,4558	1,333	+56.0	-17.82	+4.6	4. 0. 0.4	88.58	66.65	55.60	2.19	1.83	
	1,4611	1,344	+ + 28.5	-22.66	+0.33	5.0	31.10	68.90	61.13	3.09	.46	
	1,4661	1,333	+58.0	-20°24	+4.4	4 10	38.5	70.95	88	6.14 4.39	4.6	
_	1,4518	1.321	+30.1	-20.35	+1.1	4.5	35,10	64.90	53.06	5.58	টে	
	1,4499	1.817	+59.2	19.58	4.4	0.0	35.90 36.35	64.10	59.34	8.04 8.04	.05	
	1.4623	1.345	+52.0	-20.9	+1.54	000	30.65	88.5	22.02	88	61.	
	1,4672	1.364	+60.8	-23.98	+0.22	4.6	28.60	71.40	68.86	3.54	38	
	1,4568	1.888		-21.78	+1.76	00 ru	88.5	8.8 8.8	88	1.97		
_	1,4533	1.329	+47.7	-21.78	+0.26	6.2	34.45	65.55	52.33	1.28	7.4	
	1.4505	1.320	+57.4	-19.8	+4.4	3.0	35.65	64.35	58.15	8.60	98	-
	1.4593	1.337	+36.0	-14.52 	+1.54	2.0	83.8	08.10 0.10	37.95	4.12	86.	25.45
	1.4573	1.333	+59.0	-22.4	0.0	# rc	39.75	67.55	25.50	. s . c . c	9. P.	
	1.4514	1.312	+21.6	-20.05	+2.42	4.4	35.25	64.75	8.68	13.38	27	
	1.4601	1.342	+62.3	-22.0	+2.88	0.0	31.55	68.45	63.34	4.57	ক্র	- 1
	1,4550	1.334	+54.8	-19.36	+25.5	8.8	33.70	66.30	55.86	7.14	.52	
	1.4536	1.324	+37.4	-22.66	01:10	4.0	34.30	65.70	45.19	16.46	76.	
_	1.4559	1.332	+48.4	15.99	41.45	1.4	83 FD	91.89 7.99	01.4c	3.71	7.5	9.0°
_		_			) ;	1	2			20011	4	

# GROUP I. "PURE" MAPLE SIRUPS SPECIAL CHEMICAL CHARACTERS.

	Ash			Alkalini Ash		Sulphuri (SO:	e acid		
Group Number.	a. Water soluble.	b. Water insoluble.	Ratio, a/b.	Water soluble.	Water insoluble.	In syrup.	In ash.	Lead number.	Malic acid value.
	% .292 .469 .367 .379 .249 .317 .361 .363 .366 .077 .181 .394 .345 .321 .621 .382 .477 .427 .392 .185 .400 .350 .397 .246 .217	%.165 .155 .240 .150 .212 .130 .160 .185 .658 .145 .207 .160 .192 .178 .103 .200 .171 .692 .171 .172 .173	% 1.77 3.03 1.58 2.53 1.18 2.44 2.19 1.97 1.33 1.25 1.90 2.60 1.69 5.07 2.14 4.63 2.13 2.30 2.01 2.76 2.03 2.23 1.41 2.29	.425 .715 .530 .505 .125 .410 .475 .430 .445 .105 .475 .460 .435 .420 .430 .430 .435 .420 .430 .430 .430 .430 .430 .430 .430 .43	.415 .490 .550 .445 .460 .290 .445 .570 .075 .450 .510 .475 .310 .475 .245 .515 .515 .530 .425	.0104	% 1.05 2.12 1.27 1.46 1.65 1.96 1.12 1.35 2.72 1.07 1.62 2.05 2.07 1.63 1.61 .90 .48 1.38 2.26 2.47 2.47	1.36 1.52 1.45 1.49 1.42 1.32 1.56 1.45 1.45 1.33 1.28 1.37 1.08 1.41 1.46 .80 1.46 1.41 1.27 1.22 1.19	.51 .72 .34 .65 .61 .37 .76 .15 .32 .32 .52 .91 .53 .77 .57 .62 .76 .63 .63 .77 .57 .62 .76 .63 .63 .63 .63 .63 .63 .63 .63 .63 .6

### DISCUSSION

Preservatives and saccharin were not found in any case.

The solids in these sirups varied from 61.9 to 71.4%, or, excluding the lowest case, the minimum was 63.45%. Only one-third of the samples reached the national standard of 68%. Of this third, none exhibited any troublesome tendency toward crystallization. Nearly one-fourth fell below the Canada standard (65% solids).

In no case, among these twenty-five samples, was any glucose found. The percentage of invert sugar was, however, surprisingly high in some instances; as was that of the undetermined group,—acids, gums, coloring matters, etc.

Bryan noted that solutions of maple sugar inverted and heated to 87°C., gave, in most cases, a small right-hand rotation. The same fact appears in our determinations. This may be due to the conversion of a little of the easily destructible levulose to an inactive substance under the influence of the heat and of the other constituents of the solution.

The quantity of ash fell below Bryan's minimum in 7 cases, in but three of which was the deficiency very great, viz., in Group Nos. 10, 20, and 25.

The quantity of water-soluble ash fell below Bryan's minimum in the cases of Nos. 10, 11 and 20; those for insoluble ash, in Nos. 6, 10, 15, 17, 20 and 25; while the ratios between these constituents exceeded the normal limits in the cases of Nos. 15 and 17 only.

The alkalinity of the water-soluble ash fell below normal in the cases of Nos. 5 and 10, and exceeded the maximum in the cases of Nos. 2 and 15; while the alkalinity of the insoluble ash fell below the normal minimum in the cases of Nos. 6, 10, 20 and 25.

The percentage of sulphuric acid in the ash exceeded the range found by Jones in but three cases, Nos. 10, 15 and 21.

The lead numbers, for which the normal range for genuine maple sirups, is stated by Bryan as 1.19 to 2.03, in these commercial samples ran from .09 up to 1.56. The cases falling below the Bryan minimum were Nos. 10, 11 and 20.

The malic acid values found for these commercial samples ranged from .15 to .79, but were not strictly proportioned to the corresponding lead numbers. Values less than the Jones minimum were obtained in the cases of Nos. 3, 7, 10, 11, 15 and 20.

The Sy foam test has not, in our hands, given indications in close accord with those afforded by the more elaborate methods above discussed. Quantities less than 3.0 cc., Sy's minimum for genuine sirups, were observed only in cases of Nos. 11 and 17.

Giving careful consideration to the foregoing instances of departure from normal, it is clear that No. 10 is not only below standard in concentration, but that its solids exhibit at nearly every point, the characters of a mixture of a large proportion of another sugar (probably granulated sugar) with the maple sugar. In the case of No. 15, the slight deficiency in insoluble ash would, of itself, not be seriously significant, but considered with the high ratio of insoluble to soluble ash, the unusual alkalinity of the latter, the high sulphuric acid content of the ash and the low malic acid value, constitutes strong evidence of the admixture of a foreign sugar, probably brown sugar, with the maple; in the case of No. 17, appear features similar in most respects, but less convincing in degree. No. 20, like No. 10, shows pronounced evidence of admixture with a large proportion of granulated sugar; the same is true of No. 25, although the effects do not manifest themselves at so many points. No. 20 was badly fermented when the package was opened, and the fermentation may have modified the lead number and malic acid values, as well as the condition of the sugars.

## COMPOUND MAPLE SIRUPS.

Of this class of sirups, 32 samples were examined in the same particulars with the sirups purporting to be pure.

Eaton<sup>32</sup> studied the composition of mixed sirups prepared with different stated proportions of a clarified maple sirup and a plain sugar sirup, and concluded that the mixing resulted in important departures from the theory in the quantities, though not the specific alkalinity of the ash, the quantities of the lead precipitate and the Hortvet numbers obtainable, with the effect of securing upon the analysis of the mixtures indications of the presence of much smaller proportions of maple sirup than were actually present. Comparison of the several analyses of the individual ingredients and of the mixtures show, however, that the analyses of the mixtures corcomposition respond closely with those computed from the of the ingredients at all points save the Hortvet number, and accordingly indicate that the quantity and alkalinity of the ash, and the quantity of the lead subacetate precipitate afford safe guides in estimating the maximum proportion of maple sirup present in a "cane and maple" sirup, at least down to a proportion of one part of the latter to three of the cane sirup.

The description of the agent's samples follows:

## MIXED MAPLE SIRUPS

Group No.	Agent's No.	
1.	3197	Austin, Nicbols & Co., New York: Hudson Brand Syrup.* A blend made from Cane and Maple Sugar. Bought of Wm. Lender & Co., Reading, Pa. Price, —.
2.	3157	Austin, Nichols & Co., New York: Cane and Maple Syrup. A Blend made from Pure Refined Cane and Maple Sugar. Bought of Fisher Bros., Lancaster, Pa. Price, —.
3.	3188	J. H. Barker & Co., 447 W. 31st St., New York: Barker's Brand Table Syrup.*  A mixture of Rock Candy Syrup and Maple Syrup. Bought of Geo. M. Leiss & Co., Reading, Pa. Price, —.
4.	3245†	J. H. Barker & Co., 447 W. 31st St., New York: Old Time Syyrup.* This syrup is composed of the following ingredients and none other: Pure Cane and Maple Sugar. (28g.) "Guaranty: Absolutely free from Glucose, Grape Sugar, Flavoring matter and every other adulterant." Bought of Merrill & Hopper, Filbert St Philadelphia, Pa. Price, —.
5.	3155	Bay State Maple Syrup Co., Boston, Mass. Moosehead Brand Fancy—Sugar Syrup blended of Cane and Maple Sugar Syrup. Bought of Childs Grocery Co., Lancaster, Pa. Price, —.
6.	3268	Bay State Maple Syrup Co., Boston, Mass. Mt. Mansfield Brand Fancy—Sugar Syrup. Blended of Cane and Maple Sugar Syrup. H1GH GRADE MAPLE PRODUCTS. Bought of Myers, Wells & Co., York, Pa. Price, —.
7.	3151	Boyle & Williams, Bradford, Pa. Silver Seal Table Syrup. A MAPLE FLAVORED SUGAR SYRUP. Bought of Mohican Co., Wilkes-Barre, Pa. Price, —.
8.	3221	The Brooks' Green Surup, Made from Cane Sugar and Pure

Group No.	s No.	
dno	gent's	
G	4 F	
9.	3189	Fred Fear, 15 Jay St., New York: My Wife's Syrup.* A Delicious Blend of Pure Cane and Maple. Bought of J. H. Cassel, Reading. Price, —.
10.	3239	Gimbel Bros., Philadelphia. Lenox Brand, Cane and Maple Syrup. Bought of Gimbel Bros., Philadelphia, Pa. Price, —.
11.	3278	Githens, Rexamer & Co., Philadelphia, Pa. Melrose Brand Syrup. Blend Composed of Sugar Cane Syrup Flavored with Maple Syrup. (Net weight 25 oz.) (Bottled molded with name of the Towle Maple Syrup Company.)
12.	32221	C. W. Goyer & Co., Memphis, Tenn. Goyer's Maple Cane Syrup. (Device, Stalk of cane crossed by large maple leaf.) In small capitals on rear label: "Made of Pure Maple and Louisiana Cane Sugar." Bought of G. D. Williams, 721 State St., Erie, Pa. Price, —.
13.	3139	Halpen, Green & Co., Philadelphia: Apollo High Grade Vermont Style Syrup. Blended of Cane and Maple Sugar Syrup. Bought of C. W. Purse Co., Harrisburg, Pa. Price, —.
14.	3276	Halpen, Green & Co., Philadelphia: Apollo Braud Cane and Maple Syrup. (Foil Cap: High Grade Maple Products.) Bought of People's Cash Store, Gettysburg, Pa. Price, —.
15.	3192	Huntington Maple Syrup and Sugar Co., E. Providence, R. I., Golden Leaf Syrup.* Made from Refined Cane and Maple Sugar. Bought of E. W. Michael, Reading. Price, —.
16.	3270	The F. N. Johnson Co., Bellefontaine, Logan Co., O. Wild Forest Brand Syrup.* Cane and Maple. Bought of F. B. Gross, York, Pa. Price, —.
17.	3186	Francis H. Leggett & Co., New York: Nabob Cane and Maple Sugar. Paneake Syrup. Bought of A. S. Dester, Reading, Pa. Price, —.
18.	3262	Leslie, Dunham & Co., Jersey City and Newark, N. J. Green Mountain Brand Syrup.* On the rear label: Made from the Finest Selected Caue and Maple Sugars. Bought of J. Schulers, Easton, Pa. Price, —.
19.	3195	Nassau Packing Co., New York. Vermont Maple Syrup. (Sticker on main label "COMPOUND.") Bought of Cassel's Cut Rate Grocery, Penn St., Reading, Pa. Price, —.
20.	3277	New England Maple Syrup Co., Fairfax St., Johnsbury, Vt. Boston, Mass. Golden Tree Brand Fancy Quality Syrup. Made from Pure Maple and White Sugar. Bought of P. A. Miller, Gettysburg, Pa. Price, —.
21.	3142	New England Maple Syrup Co., Boston. Golden Tree Syrup.* Made from Granulated and Maple Sugars. Bought of R. J. Peters & Son, Harrisburg, Pa. Price, —.
23.	3126	Rigney & Co., Brooklyn, N. Y. Aunt Jemima's Pancake Syrup.* A Blend of Rock Candy and Maple Syrup. Bought of Z. Gray & Co., Williamsport, Pa. Price, —.
22.	3125	Philadelphia Pickling Co's Superior Vermont Syrup. Pure Maple and Cane Sugar. Bought of Dives, Pomeroy & Stewart, Harrisburg, Pa. Price,—.
24.	3275	Rigney & Co., Brooklyn, N. Y. Park Brand Maple and Rock Candy Syrup.* Bought of People's Cash Store, Gettysburg, Pa. Price, —.
25.	3140	Rigney & Co., Brooklyn, N. Y. Park Brand Syrup.* A Blend of Rock Candy and Maple Syrup. Bought of H. B. Wolbert, Harrisburg, Pa. Price, —.
26.	3209	A. B. Rogers, Elmira, N. Y. Peerless Table Syrup. Blended. Made from Maple and ?
27.	3271	Scudder Syrup Co., Chicago, Ill. Scudder's Syrup.* Pure Sugar Cane and Canada Sap-Maple and Full Measure. Absolutely Pure, Bought of R. B. Montgomery, Lewistown, Pa. Price, 25 c.
28.	3269	Thompson, 259 Greenwich St., New York. Thompson's Choice Table Syrup.* "This syrup is made from pure cane and maple sugar and preserved with 1/10 of 1 per cent. of sodium benzoate." Ou the back: "The Ingredients of this syrup are absolutely pure and wholesome the adding of granulated sugar giving richness and body, and permitting this syrup to be sold at a reasonable and not a prohibitive price." Bought of C. J. Halfrick, 240 S. George St., York, Pa. Price, 20 c.

Group No.	Agent's No.	
29.	3205†	The Towle Maple Syrup Co., St. Paul, Minn. Towle's Log Cabin Syrup (in large white letters on red ground). Cane Sugar and Maple Sugar (in small black caps on red ground). (One Pint). Bought of Cox Bros., Shenandoah, Pa. Priee, 25 e.
30.	3131	The Towle Maple Products Co., St. Paul, Minn., St. Johnsbury, Vt., Towle's Log Cabin Syrup. (Cane and Maple Sugar in small black caps on red ground. Main brand name in large white caps on red ground.) (Bottle mold bears imprint of Towle Maple Syrup Co.) Bought of S. S. Pomeroy, Harrisburg. Price, 30 e.
31.	3204	The Towle Maple Syrup Co., St. Paul, Minn. Towle's Log Cabin Sugar Syrup and Maple Syrup. Bought of Cox Bios., 9 West Centre St., Shenandoah, Pa. Price, 30 c.
32.	3201	R. C. Williams & Co., New York. Robin Hood Brand Breakfast Syrup. Selected Pure Maple and Cane Sugar. Bought of S. Davies, 21 Jordan St., Shenandoah, Pa. Price, 25 e

44

GROUP II. COMPOUND MAPLE SIRUP PHTSICAL PROPERTIES AND GENERAL COMPOSITION.

- <b>1811</b> -9901	Acids, gum, coloring ter, etc. By differ	P <sub>0</sub>	1.89	.40	2.15	1:8	0 0	. 50 . 50	0.18	0.92	5.56	1.30	16.02	195	2.20	4.52	2.35	0.33	83.51	0.35	3.03	25	1,83	8	4 0.9
	•dsA	2/2	.05	07.	11.	1.5	27.0	20.	.15	99.	60.	Ξ.8	62.	27	80	8.	.14	.13	.18	.24	.15	22	.15	11.	10
	Invert sugar.	200	4.43	2.33	98.8	1.30	07.6	1.51	2.67	2.81	8.24	1.40	20.03 8 03	3.6	3.62	9.51	5.47	15.87	27.07	3.79	1.33	5.13	7.59	7.35	00 01
•(slua	Sucrose (Olerget form	2%	57.98	62.22	85.8	8.5	5.6	60.11	64.51	. 96.19	51.86	67.34	45.94	67.26	61.20	53.63	58.04	53.48	16.89	64.32	64.49	54.38	58.46	55.74	
•(	glireeD) spilos latoT	200	64,35	65.35	67.05	8.5	8.8	65,15	67.05	65.75	65.75	70.15	02.20	69.75	67.10	00.89	00.99	70.60	67.65	68.70	69.00	62.60	68.00	64.70	01 10
	Moisture.	%	35.65	34 65	32 95	95.45 80.45	38.	34.85	32.95	34.25	34.25	29.85	39.30	30.55	32.90	32.00	34.00	29.40	38.35	31.30	31.00	37.40	32.00	35.30	000
	Sy foam test. Ce.		2.0	3.5	0.0	0.0	1.0	0.5	1.7	0.5	1.8	N -	9.0	e 60	1.9	2.4	0.7	€. Ç.	0.3	1.2	0.5	0.0	. 64 . 5	1.5	1 6
۴٠).	After inversion at Sr° C.		+0.22	+1.10	+0.22	# # C	0.0	+0.22	+4.62	+1.10	<del>ار</del> ه	9.09 1	99.04	8	0.0	0.0	+0.44	+5.28	+58.5	+0.40	+1.54	+1.33	+1.54	0.40	000
Polarization (V*)	After inversion at O. °02		-20.3	-22.1		0.06	-22.4	-50.9	-21.8	-21.78	-19.8	0 C 88	83:1	-23.1	-21.4	-22.4	-21.56	-19.8	+50.8	-24.0	-22:5	-21.12	-20.3	-22.0	21 17
Pola	Before inversion at 20° O.		+56.8	+60.5	1.83.1	155	+60.6	+58.9	+64.6	· 09+	+49.0	119.90	+59.6	+66.2	+59.8	+48.8	+55.5	+51.2	+73.2	+61.4	+63.4	+51.0	+57.2	+52.0	1 41 0 1
	Specific gravity.		1.323	1.325		1.355	1.340	1.300	1.338	1,324	1.326	1.39.	1.336	1.348	1.336	1.333	1.320	1.333	1.337	1.341	1.343	1.298	1.335	1.315	1 000 1
.17	Refractive index a C.		1.4505	1.4528	1.4543	1.4519	1.4606	1.4524	1.4568	1.4537	1.455/	1.4794	1.4583	1.4632	1.4570	1.4590	1.4543	1.4653	1.4582	1.4607	1.4615	1.4465	1.4590	1.4513	1210
-	Agent's number,		3197	3157	3245 3245	3155	3268	3151	3221	3189	37.23 30.03	3222	3139	3276	3192	3270	3186	3262	3195	3277	3142	3125	3216	25	2110
	Group Numbers.																								

8.23 2.33 2.33 2.47 8.88
diadelial:
4.48 2.75 .40 10.49 3.21 13.68 22.76
60.57 61.69 64.48 55.04 61.08 61.06
68.65 67.15 68.30 69.15 70.40 68.45 64.50
31.35 32.85 31.70 30.85 29.85 29.60 35.50
0.4+0 0.00 0.00 0.00 0.00 0.00 0.00
+1.54 -0.0 -0.0 -0.0 -0.0 -1.54 -1.56 -1.5
20.46 -22.9 -22.0 -16.5 -20.3 -22.0 -17.71
++59.0 ++59.0 ++51.0 ++61.0 +25.8
1.337 1.336 1.336 1.348 1.337 1.345
1.4606 1.4571 1.4598 1.4619 1.4648 1.4601 1.4508
3209 3271 3269 3205 3131 3204
28.57.5 28.37.5 33.4.5 33.4.5 33.4.5 33.5 33.5 33.5 3

#### GROUP II. COMPOUND MAPLE SIRUP

#### SPECIAL CHEMICAL CHARACTERS.

		As	h.		Alkalin As		Sulphur (SC	ric acid		
	Group Number.	a. Water soluble.	b. Water insoluble.	Ratio, a/b.	Water soluble. Ce.	Water insoluble. Cc.	In syrup.	In ash.	Lead number.	Malic acid. Value.
1, 2,		% .031 .352	% .018 .05	% 1.72 6.71	% .05 .44	% .07 .14	% .0027 .0061	% 5.51 1.51	.17	.20
3,		.085	.060	$\frac{1.42}{1.06}$	.13 .08	.16 .10	.0032	2.21 3.09	.27	.00
5.		.076	.022	3.40	.11	.05	.0039	3.94	.00	$.15 \\ .25$
6,		.100	.081	1.23	.15	.23	.0065	3.60	.53	.26
7,		.041	.032	1.28	.07	.10	.0039	5.27	.26	.30
3, 9.		.104	.051 $.023$	$\frac{2.04}{1.77}$	.12	.15	.0005	.32	.45	.27
۶, ),		.062	.025	2.48	.10	.06	.0029	4.46 4.94	.19	.07 .25
,		.070	.037	1.87	.11	.12	.0029	2.71	.18	.26
1		.212	.083	2.55	.29	.12	.0257	8.71	.31	.20
,		.127	.035	3.63	.16	.11	.0069	4.23	.03	.63
,		.041	.052	$\frac{4.29}{1.05}$	.28	.15	.0129	4.68 2.75	.20	.15
,		.252	.092	2.74	.29	.31	.0137	3.98	.63	.10
,		.077	.062	1.24	.08	.16	.0056	4.00	.13	.09
,		.077	.053	1.45	.10	.16	.0058	4.46	.32	.23
١,		.135	.044	3.07	.19	.12				
,		.177	.067	$\frac{2.63}{1.67}$	.21	.16	.0095	3.87	.37	.30
,		.139	.081	1.72	.12	.20	.0130	$\frac{5.08}{5.91}$	.22	.43 .46
,		.066	.074	0.89	.09	.21	.0049	3.50	.38	.14
,		.060	.050	1.20	.09	.12	.0038	3.36	.18	.25
,		.061	.072	0.85	.08	.15	.0080	5.97	.08	.19
,		.357	.060	5.95	.35	.13	.0065	1.56	.32	.21
ζ,		.077	.062	$\frac{1.24}{1.83}$	.11	.16	.0063	4.53 6.65	.20	.03
3.		.122	.105	1.16	.17	.10	.0014	.53	.20	.22
		.084	.086	.98	.13	.15	.0049	3.60	.07	.27
9		.120	.060	1.98	.17	.16	.0039	2.16	.05	.29
2.		.117	.047	2.48	.15	.13	.0043	2.61		.20

#### DISCUSSION OF ANALYTICAL RESULTS

The percentages of solids range from 62.6 to 76.3%; only 14 samples meeting the national standard requirement of 68%; 12 other samples lie between 65 and 68%.

The polarization, after inversion, and at 20°C, are negative in all but two cases, No. 12 and No. 19, which contain glucose, although its presence is not declared on the corresponding labels.

The quantities of ash, in all cases but No. 2, No. 16, and No. 26, are so low as to indicate the use of either beet sugar, granulated cane sugar, or filtered sirup from raw cane sugar as the principal dissolved sugar in the preparations. In 18 cases, the total ash does not exceed .15%, i. e., one-third of the minimum for pure maple sirup.

The proportions of sulphuric acid in the ash is excessive in 24 cases out of 32, indicating that in these cases, at least, sugars other than granulated sugar have been employed. The high sulphuric acid content of glucose shows its influence in the case of No. 12.

Most important are the indications as to the maximum quantities of maple sirup that can be present in these mixtures called "Maple and Cane," more rarely, "Cane and Maple." Taking the *lowest* proportions of the several constituents in maple, rather than the corresponding average proportions, so as to magnify the quantities of maple, and, still further, neglecting all contributions to these constituents made by the added sugars, the following proportions of maple sugar in the mixture are indicated:

By the total ash, 1/9 to 10/11;

By the insoluble ash,  $\frac{1}{8}$  to  $\frac{2}{3}$ ;

By the alkalinity of the soluble ash,  $\frac{1}{5}$  to all;

By the alkalinity of the insoluble ash,  $\frac{1}{6}$  to all;

By the lead number, none to 7/12;

By the malic acid values, none to all.

Most significant of the probable maximums is the least proportion shown by any of these properties of the sirups. They show from none in the case of No. 3, up to  $\frac{2}{5}$  in the case of No. 6, but, in 19 cases out of 32, show that the quantity of maple sirup present is, at the highest, not one-fourth of the total. In other words, most of these compound maple sirups are simply sirups made from other sugars and, at best, flavored with a little maple, if not with other imitation flavors of such nature as can not be detected by present analytical methods.

## MISBRANDING OF COMPOUND MAPLE SIRUPS

Although the declarations on the labels of these sirups in most cases made clear their compound nature, there were a number of instances in which the labels did not conform strictly to legal requirements:

- No. 12. The misleading statement, "Made of Pure Maple and Louisiana Cane Sugar" is made and the misleading device of a maple leaf and a bunch of sugar cane is used.
- No. 19. Although the word "Compound" appears in large letters, the label does not contain "a statement of the substance entering into said compound," as the law requires.
- No. 22. The word "Cane" was not printed in the manner prescribed by law.
- No. 28. Label contains the rather misleading statement, "The adding of granulated sugar giving richness and body."
- No. 29. The words "Log Cabin—Syrup," printed in white letters on red ground; the words "Cane Sugar and Maple Sugar," in small black caps on red ground, i. e., so as to be difficultly visible.

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## SUGAR-CANE PRODUCTS

From the sugar-cane (Saccharum officinarum, Linn.) comes the greatest variety of saccharin food products: Cane-syrup, molasses, brown sugar, soft white sugars, part of our granulated sugars (of which a considerable fraction is derived from crude beet-sugar,) refinery sirup—which is, properly speaking, a molasses—rock candy, rock candy sirup and other sirups made by dissolving cane sugar of some degree of refinement.

## The Sugar Cane

This plant is a member of the great Grass Family, and is, in reality, a tropical plant. It was, however, introduced into the Gulf States a century ago and has been the basis of an important series of related industries in this country. Despite its long period of acclimation in that semi-tropical portion of our land (it is grown as far north as the lower counties of South Carolina), it is still unable to produce seed, and is therefore grown from adventitious buds which develop from the internodes of the cane, when short lengths (rattoons) of the latter are planted in the furrow.

The sugar-cane is a crop of interest to our planters along the Gulf Coast from Southern Texas to South Carolina, but as well to the farmers of our dependencies, Porto Rico, Hawaii and the Philippine Islands.

## Composition of Cane Juice

The sugar is usually obtained from the cane by crushing it and expressing its juice between huge rolls. The milling process has, of late years, been improved especially by sprinkling the bagasse, or once-milled, crushed cane, which retains, like a sponge, a considerable fraction of the original juice, and putting the bagasse a second time through the rolls to recover another important fraction of the sugar held by the wetted bagasse.

The average composition of the juice expressed from the Louisiana cane is stated by Browne:

 $\begin{array}{c} \mathbf{TABI.E} \ \mathbf{XI} \\ \mathbf{Composition} \ \ \mathbf{of} \ \ \mathbf{Cane} \ \ \mathbf{Juice} \end{array}$ 

d ·	Per Cent.
Water, Ash, Nitrogenous bodies, Suerose, Dextrose, Levulose, Free acids.	1.00

	Per Cent.
Combined saids	
Combined acids, Pectin. Fat and wax,* Fiber particles,* Dirt and earthly matter.*	.15
Fat and wax *	.10
Fiber particles,*	.10
Dirt and earthly matter,*	.12
Tannin, coloring matter,	.06
	.01
	100.00

<sup>\*</sup>Mechanical impurities removed from the cane during milling.

This table shows that sugar cane juice is much richer in sugars than is maple sap. In individual cases, the percentage of sugars is much higher than the average here given.

It is noteworthy also that the cane sugar (sucrose) is here accompanied by dextrose and somewhat less levulose. When the cane is young, the latter sugars alone appear and in about equal proportions. Later, cane sugar is found, and as the season advances, the dextrose and levulose diminish as the cane sugar increases, the levulose decreasing more rapidly than the dextrose. When the cane attains full maturity, which it rarely does in the Gulf States, the levulose sometimes disappears altogether.

The nitrogenous bodies, here as in maple sap, form but a small part of the dissolved solids, although more abundant in the cane juice than in the maple sap. These nitrogenous bodies in cane juice are of great variety, as shown by the following analysis by Hardin, quoted by Browne:

Distribution of Nitrogen in Cane Juice

	juice	total
	ä	of
	entage	Percentage nitrogen.
·	Per	Perc
Nitrogen in albumen,	%	%
Mitrogen in inuciem bodies	.0039	9.4
Ntrogen in albuminoses.	.0025	6.3 5.2
	.0122	30.5
Nitrogen in amido acid amids (Asparagin),	.0098	24.0
Nitrogen in ammonia, Nitrogen in nitrates, Nitrogen in nitrogenous bases	.0024	6.1
Nitrogen in nitrogenous bases,	.0071	17.7

From the table first quoted it appears that cane juice contains also some gummy or pectinous matter. This has been found upon analysis to consist chiefly of three bodies, xylan, araban, and galactan.

Just as maple sap was found to contain an organic acid, malic acid, largely combined as the acid of malate of lime, so the cane juice too is found to contain organic acids, partly in a free state and partly in combination with mineral and possibly organic bases.

In the sugar cane, these acids are aspartic (also mentioned among the nitrogenous amido-acids), malic and succinic; while in the green cane, glycollic acid also appears. Furthermore, a small amount of a tannic acid is always present. Citric, tartaric, and aconitic acids have been reported as present in the cane, but these findings have not been confirmed.

Below is given an average of ash analyses made by Hall (quoted by Browne) of the juice of several varieties of cane:

Composition of Ash from Sugar Cane Juice

	Average
	% 45.54
Potash, K <sub>2</sub> O,	1.17 3.93 5.02
Magnesia, MgO,	.95 .54
Siliea, SiO <sub>2</sub> ,	5.72 $21.78$
Carbonic acid, CO <sub>2</sub> ,	5.00
Total, Deduct $\Theta$ =Cl,	98.6
Carbon and undetermined,	1.8
-	100.5

Alkalinity (ec N/10 per gm. ash) 29 ec.

It is understood, of course, that the proportions between the components, and as well also the total percentage of ash in the juice, will vary within rather wide limits according to the conditions of soil and fertilizer application.

Browne has noted, further, that the juices obtained from the second and third millings of the bagasse, even where no water is added to the latter, are much more impure than that obtained from the first pressing, since the ash, nitrogenous bodies and gums are much more abundant in the later pressings, although the dextrose and levulose show some decrease. The impurities (ash, etc) are also increased when the bagasse is moistened with water, and much more when it is moistened with steam, before the later millings.

The milling processes to which the cane is subjected in the sirup and sugar-making industries are very similar, but there is much difference in the succeeding steps of the manufacture of the two products, cane-sirup and cane-sugar. They will, therefore, be considered separately.

### CANE-SIRUP MANUFACTURE

The object, in this branch of the industry, is the production of a sirup, of the finest, characteristic cane-flavor, of bright color and high transparency, and of the degree of concentration best suited, on the one hand, to the highest flavor and greatest resistance of the product to destructive fermentations, and, on the other, to the maintenance of the liquid state, since over-concentration induces a tendency of the sugar to crystallize and settle as a sediment to the bottom of the container,—a condition inconvenient alike to the dealer and consumer. Since, in this manufacture, the sugar of the juice is all to be retained, cane may be utilized which, owing to the high proportion of dextrose and levulose in its juice, substances that interfere with the crystallization of the cane sugar, would be of inferior value for sugar manufacture.<sup>2</sup>

In the manufacture of the sirup, therefore, the process is often so simple as to approach the character of the maple sirup manufacture. That is, heat alone is used to clarify the juice, that is, to rid it of mechanical impurities such as bits of fiber, cane wax, etc., and of a portion of the albuminoids and germs, which rise as a scum and are either skimmed or floated off from the heated juice. In the older practice, the juice was clarified and evaporated in an open kettle set directly over the fire, a practice still in vogue where the product is intended solely for domestic use. In more modern factories, however, steam coils are used for more safely heating the juice, and the clarification and evaporation are conducted in different vessels. Browne<sup>3</sup> presents some experiments by Agee and Hall, showing the efficiency of the method for removal of much of the albuminoids and gums, but notes that the boiling in the presence of organic acids of the juice results in the conversion of considerable amounts of the cane sugar into dextrose and levulose,— a change earlier stated not to be undesirable, if not carried too far, because of its rendering the sirup less likely to crystallize on keeping. Browne states, in the same connection, that in the manufacture of sirup, clarification by heat alone is often the sole method employed. Wiley —4 notes of the manufacture by this method, that "the results have been highly satisfactory, and it can now be said, upon actual experimental data, that a table sirup can be made from the juice of sugar cane having an attractive light color and being practically free from suspended matter. In other words, the products are composed simply of the

concentrated juice of the sugar cane, from which have been removed all the substances coagulated by heat during all of the processes of concentration. Such a product can be offered to the public with absolute certainty of its wholesomeness and freedom from all injurious substances."

In many factories, however, the process of clarification follows the plan commonly used in sugar factories; that is, the juice is clarified by the use of sulphurous acid either with or without partial neutralization by lime subsequently added, heat being applied, in either case, to complete the action. The result of this treatment, if it be properly controlled is, as Browne<sup>5</sup> has shown, the more complete removal of albuminoids and gums than can be effected by heat alone and a less inversion of cane sugar. The color of the sirup is kept light and bright, but the flavor and aroma are greatly impaired by the sulphurous taste and smell which remain in the product, to say nothing of the possible injury to health which these chemical residues may cause. When the evaporation is completed in open vessels, particularly by the old kettle method, the product is entitled to the established name, "open kettle sirup." evaporated, wholly or in part, in closed vessels under reduced pressure, should not bear this name.

In view of the character of the raw material and of the processes of manufacture of cane sirup, the following standard for cane sirup has been proclaimed by the Secretary of Agriculture<sup>6</sup> under authority of Congress:

"Sugar-cane sirup is sirup made by the evaporation of the juice of the sugar-cane or by the solution of sugar-cane concrete, and contains not more than thirty (30) per cent. of water and not more than two and five-tenths (2.5) per cent. of ash."

The analytical data available from the analysis of authentic samples of cane sirup are not as numerous or complete as is desirable for the purposes of the food chemist in dealing with mixtures of canesirup with other similar materials.

Analysis made by the Department of Agriculture of sixty-nine cane sirups made in Georgia in 1903 and 1904, show the following average and range of percentages for the more commonly determined constituents:

Analyses of Georgia Cane-Sirups (Per Cent.)

Analyses of coorses					
	Moisture	Solids	Cane-sugar	Dextrose and levulose	Ash
Average, Highest, Lowest,	24.16 21.50 31.13	75.84 78.50 68.87	48.67 63.70 25.30	20.88 48.08 6.70	.98 1.61 .52

### MOLASSES

Molasses (Cane-molasses) is the liquid residue obtained when the cane-sugar in clarified, concentrated, cane-juice is crystallized out and separated from the liquid. Beet molasses is the corresponding product from the sugar beet, but possesses such flavor and odor as to make it unpalatable for human consumption.

The molasses is the secondary product, the sugar being the more valuable substance. Since every accompanying substance dissolved in the cane juice, acts in greater or less degree to hold back from crystallization a portion of the cane-sugar, it is the sugar-maker's object to remove these substances as much as practicable before concentrating the juice to the point at which the sugar crystallizes out.

The composition of molasses varies both with that of the cane-juice from which it is derived, with the character of the processes used for sugar-making, and with the degree to which the cane-sugar is removed from the treated juice. The effects upon the molasses of the treatments adopted for purifying the juice, are regarded as of secondary importance.

A number of processes have been used, in addition to simple heating and removal of the scums thus brought to the top. Formerly lime alone was added to make the liquid alkaline; but this process has the disadvantage of causing not only a loss of sugar by inversion, but the formation of black, bitter, lime dextrosate and levulosate, which impair the quality of both the raw sugar and the molasses. In most sugar-factories, the sulphitation process is used, the sulphurous acid being introduced formerly as gas, more recently in the form of solid acid sulphite of lime.

An excess of sulphurous acid is thus introduced and the juice then brought back to weak acidity or to neutrality before heat is applied. The result is not only the removal of a large fraction of the impurities left behind, as scums and as filter-press cake upon the skimming of the hot juice and its subsequent filtration, but also the avoiding of losses by sugar inversion and the bleaching of the juice; so that filtration through bone-black, formerly used to decolorize the juice before concentration, is now usually omitted. The sulphurous acid exists in the juice partially free, partially combined; and part of it is volatilized in the later concentration of the juice and part oxidized to sulphuric acid, but some of it remains unchanged.

To avoid the effects of this undesirable residue, some manufacturers have tried the use of lime followed by concentrated phosphoric

acid. The cost of the latter reagent has prevented its general use, and also the difficulties resulting from its separation as insoluble lime phosphate before the recovery of the sugar from the molasses has been carried to the usual degree of completion. Still another method, though not much used in cane-sugar manufacture, is what is known as the "carbonatation" method, in which, after the liming, carbonic acid gas is run through the liquor to take out the excess of lime. The result, however, is to increase the "gums" present in the clarified juice.

In the older methods of manufacture, the purified juice was concentrated in open-kettles, and when the mass had grained, owing to the crystallization of part of the sugar, the sugar was cured, or purified by draining and washing off the adherent molasses. Owing to the high concentration of the juice in the latter stages of this process, the boiling temperatures were high and resulted in the inversion of considerable sugar, and, through the effects of the invert sugar in preventing the crystallization of the cane-sugar, in an additional sugar loss for the latter reason.

The old fashioned "New Orleans" molasses, with its high content of cane-sugar, low ash, and distinctive flavor, was made chiefly in the manner just described.

Today, however, vacuum apparatus of various kinds is employed to make possible the concentration of the jnice at low boiling temperatures with a consequent diminution of the sugar inversion. Moreover, the molasses is re-treated one or more times to recover, so far as practicable, the cane-sugar it retains after the first crystallization. The separation of the molasses is now effected in powerful centrifugals, in which the sugar crystals are washed. The sugar produced in the better equipped factories is not the dark brown Muscovado of half a century ago, but a soft, almost white product.

The molasses, on the other hand, consists more largely of invert sugar, and other impurities that escaped removal during the clarification, or were formed during the subsequent processes. Consequently the flavors and other properties are distinctly different from and inferior to those of the older cane molasses. The most impure molasses is gummy, bitter, dark colored and rich in ash, and is commonly known as "black strap." Its sugars fit it for use in the fermentation industries, and, with proper management, it is valuable as a cattle-food. Owing, however, to its high content in laxative salts and for other reasons, it is not a desirable table article.

Browne<sup>7</sup> gives the following analysis as representative of the various molasses produced by the modern methods, from a juice of average quality:

	First molasses	Second molasses	Third molasses
Cane sugar, Dextrose, Levulose, Albuminoids, Amids, etc., Acids, gums, etc.,	% 53.60 8.76 8.00 .20 .94 4.50 4.00	% 41.70 12.20 12.50 .25 1.50 6.50 5.35	% 31.70 15.00 16.50 .33 2.00 8.20 6.30
Total solids in product,	80.00	80.00	80.00

Wiley<sup>8</sup> published, in 1892, the results of a large number of analyses made under his direction by experienced analysts in different parts of the Union. From his data the writer has compiled the following summary:

Description	No. of sample	Moisture.	Solids	Sucrose	Reducing sugars	Ash	Undetermined
New Orleans molasses Average, Highest, Lowest,		25.04 35.50 16.70	74.96 83.30 64.50	42.01 66.00 16.40	23.01 41.0 19.6	3.32 8.90 .69	6.62 $24.23$ $-8.10$
lb., samples taken in Louisiana: Average, Highest, Lowest,		26.55 31.20 22.90	73.45 77.10 68.80	39.61 56.8 28.9	26.25 37.8 17.0	3.46 6.28 .85	4.13 8.04 .37
"Open-kettle" New Orleans molasses: Average, Highest, Lowest,		23.95 28.69 17.17	76.05 82.83 71.31	49.97 59.07 39.23	19.91 28.98 14.58	2.11 3.15 .85	4.06
Porto Rico molasses: Average, Highest, Lowest,		25.79 29.85 21.28	74.21 78.72 70.15	39.44 50.08 21.78	21.82 30.00 14.36	3.08 5.33 1.00	9.87
Black strap molasses:  Average,  Highest,  Lowest		22.80 29.14 19.30	77.20 80.70 70.86	27.02 36.97 7.92	29.96 54.05 17.21		12.11

Analysis by Babington<sup>9</sup> of 31 samples of modern molasses, show the following range of composition:

	Per cent.
Cane sugar, Reducing sugars, Ash. Moisture,	32—52 13—24 0.5— 4 29—32

The United States Standard<sup>10</sup> for molasses is:

"Molasses is the product left after separating the sugar from masseuite, melada, mush sugar, or concrete, and contains not more than twenty-five (25) per cent. of water and not more than five (5) per cent. of ash."

Browne<sup>7</sup> observed that the molasses of lower Louisiana showed far more chlorides than those of the upper portions of the State, and observes that the former are, for that reason, less desirable for table use because of the influence of these salts upon the flavor.

It is further to be noted that molasses is a fermentable material. The fermentation usually begins at the surface, which is doubtless diluted by absorbing atmospheric moisture, but, ere long, it affects the entire mass. For this reason, modern molasses-packing establishments commonly sterilize the product and deliver it hot into the tin vessels commonly used as containers for the retail trade of today.

#### REFINED MOLASSES

The ordinary cane molasses possess little of the flavor and aroma characteristic of the fresh cane and of the old time open kettle sirup. The substances to which these characteristic qualities are due are apparently not destroyed, but simply "bound" chemi-The color of the molasses too is unattractive. This product is, therefore, sometimes subjected to so-called "refining processes," the products from which are termed "refined molasses." processes are often quite crude. In one method, a suspension, in water, of acid sulphite of sodium is brought into contact with zinc dust, and, after these have reacted for a few moments, the resulting solution is strained through a cheese cloth and then mixed, by means of a stirring apparatus, with the crude molasses. A vigorous effervescence follows, with a bleaching of the molasses and a liberation of the characteristic cane aroma and flavor, although both are obscured by the accompanying effects of free sulphrous acid. In some cases Whichever a treatment with oxalic acid.11 these courses is pursued, there is danger that the resulting "refined" molasses may contain small amounts of a poisonous substance, zinc salts or oxalic acid salts.

In other cases, the refining consists simply in a removal of part of the color by filtration through bone-char, or in a bleaching by aid of ozone, hydrogen peroxide, sulphurous acid, sulphuric acid, or chloride of tin.

The common forms of adulteration of these products other than by the introduction, in the course of treatment, of injurious substances, such as zinc, tin, and sometimes copper, (and very serious consideration must be given in this connection to the residual sulphurous acid), is by the substitution of glucose for a part of the sugar cane product. Misbranding also is not unknown, such as the designation as "open kettle," of products obtained by other processes. Concerning the signification of the qualification "New Orleans" used in conjunction with the word "molasses," the Board of Food and Drug Inspection of the U. S. Bureau of Chemistry, by the vote of two of its three members, decided that its original signification of open kettle drippings or "bleedings" has disappeared, but that it should be restricted to molasses produced in Louisiana.<sup>12</sup>

#### MOLASSES SAMPLES ANALYZED

The following samples, classified according as they bore the names, cane sirup, molasses, New Orleans molasses, and refined molasses, respectively, were submitted by the special agents of the Bureau:

Group No.	Agent's No.	
		Sugar Cane Sirup.
1.	3241	E. C. Joullian Canning Co., St. Bernard Parish, La. St. Bernard Brand Sugar Cane Syrup. Bought of Gimbel Bros., Philadelphia, Pa. Price, 20 c.
		Molasses.
Ι,	3128	P. Duff & Sons, Pittsburg, Pa Palmetto Brand Molasses for Baking and Table Use. Contains Sulphur Dioxide. Bought of D. Pollock, Harrisburg, Pa.
2.	3225	P. Duff & Sons, Pittsburg, Pa., Red Lion Style Louisiana Baking Molasses. Contains Sulphur Dioxide. Known for its baking qualities and rich cherry color. Bought of Blase Bros., Erie, Pa.
3.	3215	P. Duff & Sons, Pittsburg, Pa. St Catharine Brand Molasses. For Baking. Contains Sulphur Dioxide. Bought from C. W. Hicks, Williamsport, Pa.
4.	3233	Hearn & Jones, New Orleans, La. Rockwood Pure Molasses.* Contains Sulphur Dioxide. Bought of Mr. Dwyer, Pittsburg, Pa.
5.	3232	Hearn & Jones, New Orleans, La. Woman's Club Brand Pure Molasses.* Contains Sulphur Dioxide. Bought of Kauffmann's, Pittsburg, Pa.
6.	3219	Penick & Ford, Ltd., New Orleans and Shreveport, La. Orla Brand Old Fashion Type Molasses.* Contains Sulphur Dioxide. Absolutely pure as it comes from the plantation. Bought of D. F. Detrick, Williamsport, Pa.
7.	3264	S. Scheiver & Bro., Paterson, N. J. Premium Molasses. Contains Sulphur Dioxide. Stick label: Preserved by use of one-tenth of 1 per cent. Benzoate of Soda. Bought of S. Schuyler, Easton, Pa.
8.	3258	R. C. Williams & Co., Distributors, New York. Coronet Brand Molasses.* Absolutely Pure and Especially Prepared for Table and Baking Purposes. Contains Sulphur Dioxide. Bought of Mohican Co., S. Front St., Easton, Pa.

Group No.	Agent's No.	
	1	New Orleans Molasses.
1.	3227	N. H. Alexander Co., Cincinnati, O. Cherry Grove Pure Cane Dark Red New Orleans Molasses. Warranted Pure and Wholesome. Contains Sulphur Dioxide. Bought of Mrs. Drauz, Erie, Pa.
2.	3224	N. H. Alexander Co., Cincinnati, O. Robin Red Breast Brand Pure Red New Orleans Molasses, Pure and Wholesome. Contains Sulphur Dioxide. For Baking and Table Use. Bought of Jno. Scarlett Co., Erie, Pa.
3.	3231	N. H. Alexander Co., Cincinnati, O. Tiger Brand Pure New Orleans Molasses. None Better for Any Purpose. Contains Sulphur Dioxide. Choice light color. Bought of Kauffmann's, Pittsburg, Pa.
4.	3252	Brand' New Orleans Molasses. Contains Sulphur Dioxide. Bought of G. M. Dunlap Co., Chester, Pa.
5.	3251	Central Molasses and Syrup Refinery, Philadelphia, Pa. Dunlap's 10 c. New Orleans Molasses. For Baking and General Use. Packed for Geo. M. Dunlap Co. Contains Sulphur Dioxide. Bought of Geo. M. Dunlap, Chester, Pa.
б.	3220	Clark, Chapin & Bushnell, New York. Orange Grove Pure New Orleans Molasses. Warranted Pure and Wholesome. Contains Sulphur Dioxide. Bought of D. F. Dietrick, Williamsport, Pa.
7.	3146	P. Duff & Sons, Pittsburg, Pa. Duff's New Orleans Molasses (No. 2 1/3). A High Grade Product to which we have added Louisiana Sugar, which increases its sweetness and value. Contains Sulphur Dioxide. Device, Ginger Bread Man. Bought of Jonas Long's Sons, Scranton, Pa.
8.	3154	Hearn & Jones, New Orleans, La., Distributors. Old Mill Brand Pure New Orleans Molasses.* Contains Sulphur Dioxide. Bought of Childs Grocery Co., Lancaster, Pa.
9.	3250	Hearn & Jones, New Orleans, La. Pure New Orleans Molasses, Sunflower Brand.* Always Uniform, of Special Fine Flavor. Selected (for) Table and Baking Quality. Contains Sulphur Dioxide. Bought of Columbian Tea House, Chester, Pa.
10.	3238	James W. Houston Co., Pittsburg, Pa. Willard Brand New Orleans Molasses. For the Best Family Trade. Excellent for Baking. Contains Sulphur Dioxide. Bought of Chas. Bergman, Pittsburg,, Pa.
11.	3191	Penick & Ford, Ltd., New Orleans and Shrevcport, La. Aunt Diuah Brand Pure New Orleans Molasses.* Bought of N. A. Flickinger, Reading, Pa.
12.	3150	Penick & Ford, Ltd., New Orleans and Shreveport, La. Brer Rabbit Brand Pure New Orleans Molasses.* This Molasses is Selected for its Exceptional Baking Qualities. Contains Sulphur Dioxide. Bought of Mohican Co., Wilkes-Barre, Pa.
13.	3218	Steuart, Knatz & Co., Baltimore, Md. Old Time New Orleans Molasses. For Table and Baking. Crown Brand. Quality Guarauteed. Contains Sulphur Dioxide. Bought of Z. Gray & Co., now J. H. Bertin & Co., Williamsport, Pa.
14.	. 3211	The Winter Ousen New Orleans Molasses.*
		Refined Molasses.
1	3226	P. Duff & Sons, Pittsburg, Pa. The Only Molasses Refiners. Duff's Refined Molasses. The Best for Every Purpose. Flavor, Baking, Streugth, Sweetness, Cheapest. Guaranteed the richest and finest flavored for table use. Made from high grade New Orleans Molasses, with all dirt, chemical and vegetable impurities removed. Bought of Mrs. Drauz, Erie, Pa.

#### ANALYTICAL EXAMINATION.

The following determinations were made on these samples:

#### Physical:

a. Refractive index (28° C.)

Specific gravity.

Polarization, direct (20° C.) Polarization, indirect, (20° and 87° C.)

#### Chemical:

Reducing sugars.

- Ash, total. Ash, water soluble. Ash, water insoluble.
- Alkalinity of soluble ash, (from 1 gram of sample). Alkalinity of insoluble ash, (from b gram of sample.)

Sulphuric acid (SO3).

I. Sulphurous acid (Steam distillation).

Zinc. m.

- Tin. n.
- Benzoic and salicylic acid, and saccharin.

These determinations were made according to the official methods, except those of tin and zinc.\*

The results of these determinations are given in the following tables:

weighed as zinc oxid (ZnO).

<sup>\*</sup>Determination of Tin and Zinc: The method used was as follows: 50 grams of the molasses was ignited to a gray ash in a porcelain dish with 15 cc. concentrated sulphuric acid. The ash was then dissolved by boiling for 30 minutes with concentrated hydrochloric acid, the solution diluted with water, filtered, the residue on the filter well washed. The united filtrate and washings were nearly neutralized with ammonia water, hydrogen sulphid passed through the liquid, and the latter allowed to stand in a warm place over night. The precipitated sulphids were filtered, and washed on the filter; after which, the tin and zinc sulphids were dissolved out by passing boiling ammonium sulphid through the filter. In a few cases, marked \* in the table, a little black, lead sulphid was left undissolved. The tin sulphid was separated by acidulating the filtrate, the precipitate filtered off, washed, ignited and weighed as tin oxid (SnO<sub>2</sub>). The filtrate and washings from the tin sulphid were treated at boiling heat with bromin to oxidize ferous salts, the excess of bromin boiled off, ferric chlorid added until a yellow color appeared, the liquid nearly neutralized with ammonia water, a little acctic acid added, and the liquid boiled. The zinc sulphid, thereupon separated, was filtered off, washed, ignited and weighed as zinc oxid (ZnO).

			(0)	Pola	Polarization (V°)	<u> </u>			(:			
Group. No.	Agent's No.	Sp. gr. (60° F)	Refractive index (28°	Direct (20° C)	(O °02) toerfbul	(O .18)	Moisture	Solids (Geerlig)	Cane sugar (Clerget	sıszus zaisubəH	dsA	$U_{ m B}$ determlned
Shear-cane Syrub,	3241	1.411	1.4858	+24.4	-20.0	+0.9	29.00	71.00	56.08	8.72	1.91	3.29
Molasses (Other than New Orleans)—  2, 3, 4, 5, 6, 7,	3128 3225 3225 3235 3233 3232 3232 3264 3264	1.389 1.357 1.390 1.391 1.391 1.387 1.408	1.4734 1.4763 1.4754 1.4774 1.4777 1.4768 1.4936 1.4936 1.4936	+ 441.4 + 31.6 + 24.4 + 37.6 + 40.4 + 33.6 + 30.0	-18.48 -11.90 -16.7 -20.0 -17.6	++ 0.0 0.0 1.3.5 0.0 0.0 0.0 0.0 0.0 0.0	26.15 24.95 24.95 24.60 24.75 18.20 28.80 23.35	73.85 74.65 75.05 75.40 75.20 75.20 76.65	47.85 32.79 29.02 35.22 43.42 43.72 38.60 35.60	23.08 15.28 12.42 22.30 22.30 22.30 22.34	5.7.7.51 5.7.7.51 5.38 5.44 5.44 7.7.7.51	. 37 19.07 26.03 12.82 6.09 18.52 9.44 12.94
New Orleans Molasses—  1, 2, 3, 4, 5, 6, 7, 10, 11, 11, 12, 13, 14,	3227 3221 3231 3231 3251 3250 3144 3154 3256 3188 3189 3218	1.410 1.365 1.407 1.406 Fermented 1.406 1.350 1.350 1.385 1.385 1.385 1.385 1.385 1.400 Fermented 1.385 1.400 Fermented 1.400	1.4881 1.4793 1.4793 1.4784 1.4802 1.4784 1.4784 1.4735 1.4735 1.4735 1.4735 1.4735 1.4735 1.4735 1.4735 1.4735 1.4735 1.4735 1.4735 1.4735 1.4783	++++++++++++++++++++++++++++++++++++++	16.77 17.27 17.23 18.00 18.00 17.00 17.00 17.00 17.00 18.55	++++++++++++++++++++++++++++++++++++++	88888888888888888888888888888888888888	77.00 76.25 74.35 74.35 76.00 75.00 75.85 73.95 74.70 77.05	29.17 29.16 29.17 29.28 29.28 29.28 29.28 29.29 29.33 20.33	15,42 22,94 15,14 15,14 11,14 11,15	7.00 4.00 4.00 4.00 4.00 4.00 4.00 4.00	14.81 20.62 8.99 15.24 16.00 18.52 11.33 10.47 11.72 11.72 11.72 13.35
Refined Molasses-	0.00	200	1 (706	9	17.6	80 0+	23.65	76.35	44.02	17.56	4.29	10.48

#### SUGAR-CANE PRODUCTS

#### II. Special Chemical Characters

			-	Alkali	nity.	(SO <sub>3</sub> ) in	(SO <sub>2</sub> ) in		
Group Number	Agent's number.	Soluble ash.	Insoluble ash.	Soluble ash.	Insoluble ash.	Sulphuric acid (SC molasses.	Sulphurous acid (Someolesses.	Zine in molasses.	Tin in molasses.
Sugar-cane Syrup.									
1,	3241	% 1.540	$\frac{\%}{0.370}$	Ce. 1.58	Cc. 0.92	% 1.372	P.p. Mill. 32	P.p. Mill. Trace.	P.p. Mill. 18.9
Molasses (other than New Orleans).	1								
1,	3128 3225 3215 3233 3232 3219 3264 3258	4.056 6.335 5.220 4.160 2.764 2.700 3.836 4.750	0.945 1.175 2.360 1.140 0.680 0.740 1.150 1.020	2.77 4.38 2.88 3.42 2.14 2.12 2.70 1.64	2.12 3.26 5.22 2.54 1.40 1.60 2.66 2.42	.5109 .2352 .49049 .5365 .3615 .3162 .4411 .4596	32 50 35 70 32 92 188 80	None. 32.1 14.5 † Trace. 9.6 20.9 35.3	23.6 23.6 39. 28.4 44.1 14.5 28.3
New Orleans Molasses.									
1,	3227 3224 3231 3252 3251 3220 3146 3154 3250 3238 3191 3150 3218 3211	5.768 5.280 3.410 3.926 4.460 4.530 3.490 3.650 4.274 3.550 3.255 3.860 3.230 4.422	1.832 1.400 1.530 0.914 1.380 1.255 0.750 1.082 0.676 1.170 1.205 0.910 0.720 1.530	2.86 3.73 2.32 4.08 0.80 2.74 1.97 3.07 1.90 3.11 2.88 2.88	4.48 3.12 3.66 2.26 3.54 2.92 1.67 2.48 1.74 2.38 3.03 2.12 1.44 3.36	.7210 .3189 .4960 .4390 .3670 .5683 .3474 .4054 .3211 .4610 .5086 .4476 .2916 .6380	51 64 902 80 32 61 112 80 54 86 49 0 45 57	9.6 12.8 † Trace. 45.0 None. 25.7 17.7 16.1 22.5 14.5 None.	42.6 17.3 *37.9 † 17.3 25.2 None. 26.8 15.8 22.1 28.4 *22.1 None.
Refined Molasses.									
1,	3226	3.532	0.760	2.57	1.67	.5230	45	19.3	29.9

†Not determined. \*Contained some lead.

## Discussion of Analytical Results

The sugar cane sirup, No. 3241, corresponds well in the general composition of its solids, with average cane sirup. Its concentration, however, is too low.

The molasses samples show, in general, a good measure of conformity to the national standard for solids; only two samples, out of the 22 examined, show a deficiency of more than 1% from this standard.

As respects their ash content, however, the conditions are not so good; 11 samples, or one-half of the total number examined, having ash more than one-half per cent. in excess of the standard maximum.

This character points to a lower grade in the quality of these products than is desirable for table or cooking purposes. It is interesting, in this connection, to observe that the food standards proclaimed by the Louisiana State Board of Health<sup>13</sup> follow quite generally the national standards, but increase the ash maximum for molasses from 5 to 10 per cent.; and for cane sirup, from 2.5 to 4.2 per cent.; while for the molasses, the water maximum is increased from 25 to 30 per cent.

Three other qualities of these samples point in the same direction: The deep black color many of them show, the high proportion of reducing sugars to the cane sugar (sucrose) present, and the large proportion of undetermined matters. The evidence furnished by the analyses with respect to these four characters, points to the sale for human consumption, of a large quantity of "black strap" unsuited for such use.

It is only fair, in this connection, to note the serious difficulties encountered in making accurate analyses of molasses, which, by reason of their sticky, viscous condition, are not readily weighed out, dissolved, and filtered, and because of the dark color of their solutions, are very hard to polarize with exactness. The general tendency is probably to read the polarizations too low, and thus to understate in the analysis the percentage of cane sugar present, and to overstate the "undetermined matters."

The polarizations at 87°C, after inversion of the molasses solutions, exhibit a general tendency toward dextro-rotation, instead of the neutrality which theoretically should appear. The degree of dextro-rotation, in those cases not marked\* because of the very dark color of the solution, is, however, too small to suggest any cause other than a partial destruction of the levulose by the heating method employed.

In other words, these samples do not exhibit the prevalent adulteration with glucose that appeared, thirty years ago, at the time of Wiley's investigation of the molasses on the retail market.

The tabulated analyses justify somewhat more extended discussion at certain points:

Ash: The quantity of ash in the 22 samples of molasses (group 2 and 3) range from 3.44 to 7.60 per cent., and average 5.29 per cent.

The quality of the ash varies not only with that of the plant as influenced by soil and fertilizer treatment, but also with the manufacturing operations.

The samples show wide differences at a number of points. For example, the *solubility* of the ash in hot water: The average percentage of water-soluble ash was 4.129 per cent., equivalent to 78 per cent. of the total ash; but the proportion of the ash that is water soluble varies, in the different samples, from 68 to 85 per cent. Com-

parison with the proportions in this respect exhibited by samples of known history, is impossible because few figures are available in the literature on the subject.

Again, taking the percentage of sulphuric acid (SO<sub>3</sub>) in the ash into consideration, it is clear that the amount of sulphuring and the degree to which the injected sulphurous acid is oxidized to sulphuric acid, must affect the proportion of this constituent appearing in the ash. Browne<sup>14</sup> states that in the ash of typical molasses made by mill sulphitation, the usual method, he found 10.79 per cent. of SO<sub>3</sub>; in that from diffusion sulphitation 6.72 per cent; from the open kettle process, 10.94 per cent.; and rather curiously, from carbonatation, 11.18 per cent. In the commercial samples we examined, the quantity of SO<sub>3</sub> in the ash, stated in terms of the entire molasses, ranged from .2352 per cent. to .7210 per cent., average, .4401 per cent. In terms of the ash itself, the quantities ranged from 3.13 to 11.42 per cent., average 8.50 per cent of the total ash. The low percentages in samples 3225 (3.13%) and 3224 (4.88%) are the only notable figures in the list.

Sulphurous acid: The reasons for the presence of this chemical reagent have already been set forth. It is clear that this constituent is not used by molasses makers for the preservation of their product, but that it has been in use for many years primarily as a clarifying agent, secondarily as a decolorizer. The researches of the Louisiana Sugar Experiment Station<sup>15</sup> failed to show any free sulphurous acid in Louisiana molasses. It is well known that this acid has a very marked tendency to unite with some saccharin substances to form compounds that do not liberate their sulphurous acid content when brought into contact with weak organic acids, but they do slowly and continuously liberate their sulphurous acid even at ordinary temperatures in contact with dilute mineral acid, such as the hydrochloric acid of the gastric juice.

The quantities appearing in these samples range from none to 902 parts per million of molasses; average, 102 parts per million.

The Food Act of May 13, 1910, provides, "That in the preparation of dried fruits and molasses, sulphur dioxide, either free or in simple combination, may be used in such quantities as will not render said dried fruits or molasses deleterious to health." The danger point is not specifically indicated. Wiley, 16 after an experiment of twenty days, during which sulphurous acid was fed in the form of sodium sulphite and of a water solution of gaseous sulphurous acid, in amount ranging from .078 to 1.020 grams each day for each subject, concluded that in these quantities the sulphurous acid is decidedly injurious to health; on the other hand, Blouin and his associates, 15 in experiments in which from .084 to 0.299 grams of sulphurous acid were administered daily in molasses and can sirup to healthy ne-

groes, found no indications of injury to health at certain of the points where injurious effects were indicated by Wiley's experiments. The sirup and molasses used by the Louisiana experimenters contained, according to their report, sulphurous acid as follows, in parts per million of the sirup, etc.: Sirup, 910, open kettle molasses, 1st sample, 322; 2nd sample, 1004; vacuum process molasses, 1856\*.

However satisfactory to manufacturers these conclusions from the Louisiana experiments may be, consumers will welcome the day when the present sulphitation process is replaced by some other method, practicable to the sugar-maker and beyond objection by the consumer.

Of the 18 molasses samples examined for this poisonous Zinc: metal, all but 4 were found to contain it in amounts varying from mere traces to 45 parts per million of molasses, average, 15.3 parts. It is not clear how these quantities of zinc find their way into the molasses. Gladstone<sup>17</sup> found that zinc, like copper, is but slightly attacked by sugars; although, as Salkowski<sup>18</sup> and Fileti<sup>19</sup> found that dextrose attacks copper oxid, it may be that zinc oxid also would yield to the action of invert sugar. As a matter of fact, however. galvanized vessels are little, if at all, in use in the sugar-house. A more probable source of this impurity is the zinc chlorid commonly used as a flux for the soldering of the tin cans in which the molasses is commonly put up for the retail market. Unless the cans are thoroughly washed before they are filled, zinc would be thus introduced, where zinc chlorid is used in place of rosin, for a flux.<sup>20</sup>

Tin: Of the 20 samples examined for this poisonous metal, all but two were found to contain it in quantities ranging from 11.0 to 42.6 parts per million, and, in several cases, it was accompanied by a little lead. The average quantity found was 23.4 parts per million.

In this case, it seems improbable that the tin was introduced in the course of manufacture, since the use of tin salts to impart a yellow color to certain grades of sugar, is probably not practiced in Louisiana at the present time. Molasses is, however, a distinctly acid product and may, by the action of its acids, dissolve some of the tin from the inside walls of the tin containers commonly used. It should be said, however, that the walls of the cans showed, at the time when the containers were opened and the portions of samples taken for analysis, no strong evidence of such attack.

Of the two metals together, the samples examined for both contained from 23.6 to 70.2 parts per million. Weber<sup>21</sup> found from a trace to 640 parts per million in 16 samples, average 132 parts. The quantities we have found are, therefore, well within the range of experience.

<sup>\*</sup>These figures are obtained by multiplying by 2 the original results, which are expressed in terms of sulphur, not sulphurous acid.

It is deserving of mention that the Louisiana food regulations specifically bar the sale of sirups and molasses containing these metals.

Refined molasses: Of these products but one sample was submitted The method used in refining is unknown. its general composition nor in its special chemical characters, does it fall outside the range of ordinary molasses.

## Misbranding

The Food Act of May 13, 1909, in addition to its prohibition of deceptive labelling specifically provides that "when——any quantity of sulphur dioxide is used in the preparation of——molasses, the fact that ———— sulphur dioxide has been used in the preparation thereof shall be plainly stated on the label."

The legal tolerance for sulphurous acid does not extend to any sugar product except molasses.

The labels, in all but two cases, contain a declaration of the presence of sulphur dioxide, usually in type as large as the law specifies; but usually the declaration fails to be made plain to the purchaser. For example, attention is called to the place and manner of declaration in some of the cases:

- Obscure, against a dark background.
- Very obscure.
- 3258: In black letters on red ground.
- 3233: Small letters on side label.
  3146: In fine type on edge of label; also 3231 and 3238.
  3211: On side label, at bottom.
- In faint color.
  In very small letters.

It is impossible, with the present analytical data at least, to determine the truth of the label statements concerning the places of production of these products. The analyses indicate clearly, however, that the seller's enthusiasm (?) rather than the real qualities of the commodities account for a large fraction of the superlatives used on these labels.

## Sugar Refining Products and Sugar Sirups

The raw sugars formerly made carried upon their surfaces considerable quantities of molasses not removable by the curing processes then practiced. Today, however, the molasses is much more perfectly Browne<sup>22</sup> says, "In Louisiana a very pure white sugar is made by spraying with several sprinklings of water (in the centrifugal); such sugar is over 99 per cent. pure sucrose, the remainder being mostly moisture. In Cuba and Porto Rico they aim to make a 96 per cent. sugar. In Hawaii and Java, a sugar testing 97 per cent. is desired."

The raw sugars are put through careful refining processes in order to obtain the hard-grained, white, granulated, loaf, and flour sugars. The process is briefly as follows: The raw sugar is dissolved by water and steam, the solutions strained, then defecated, not by lime as in the case of the caue juice, but by beef's blood, with or without bone black. The albumen of the blood is coagulated by heat, like white of egg when used to clear coffee, and gathers into its mass the fine suspended impurities of the sugar solution. If the molasses is very acid, lime sufficient to neutralize it may be used, and later removed by phosphoric acid, the phosphate of lime assisting in the clarification. The liquid is then run off through thick cotton filters, through which it passes as a brown to brownish liquor, which is then re-filtered through bone black to decolorize it. The first portions of liquid coming through the filter are white; the latter portions, however, still retain a little color and are run to a separate tank, from which they are drawn to make "soft" sugar, while the colorless solution is used for the finest sugars. The concentration of the decolorized sirup follows, vacuum apparatus being exclusively used, and the method of conducting the operation determining the production of "hard" or of "soft" sugar. After crystallization is completed, the refiner's molasses is removed by the use of centrifugals of one form or another.

The amount of impurity in these molasses differs, of course, with the quality of the raw sugar used and with the completeness of the purification which its solution has undergone in the refining processes. Different trade names are applied to these products:

"Green sirup is the sirup centrifugated from the second products in the refining process."

"Golden sirup is produced from a refiner's molasses by diluting, filtering through bone black and then concentrating."

"Treacle is a name formerly given to the drainings from the dark molasses sugars called bastards,"23

The sirupy liquors obtained in the last stages of the refining of beet sugars are also used for table sirups, the crystallization of the cane sugar being prevented by its partial inversion.<sup>24</sup>

In addition to these secondary products of the sugar refinery, there are other products, more properly *sirups*, made by the solution of sugars, and also the mother liquor left from the manufacture of rock candy and known as "rock candy sirup." The proportions of impurities in these products vary with the material used in their preparation.

The general nature of the principal classes of this group is shown by the following summary:

	Water	Sucrosc	Glucose	Ash	Organic impurities	Analyst
Green syrup,	27.7 22.7 19.98 23.4 28_56	62.7 39.6 42.76 32.5 53.94	8.0 $33.0$ $26.41$ $37.2$ $9.20$	1.0 2.5 4.51 3.5	0.6 2.8 6.34 3.5 7.93	Wallace <sup>25</sup> Wallace <sup>26</sup> Wiley <sup>8</sup> Wallacc <sup>25</sup> Wilcy <sup>8</sup>

The national standards for refinery molasses or sirup and sugar sirup<sup>26</sup> are:

"Refiner's sirup, treacle, is the residual liquid product obtained in the process of refining raw sugars and contains not more than twenty-five (25) per cent. of water and not more than eight (8) per cent. of ash."

"Sugar sirup is the product made by dissolving sugar to the consistence of a sirup and contains not more than thirty-five (35) per cent, of water."

The samples belonging to this group of products submitted by the sampling agent were:

#### REFINER'S MOLASSES AND SUGAR STRUP ANALYZED

The following samples, including some that are apparently imitations of maple sirup, though not sold under indicative names, were sumbitted by official agents.

Group No.	Sample No.	
1.	3249	E. Bradford Clarke Co., Ltd., 1520 Chestnut St., Philadelphia, Pa. Pure and Unadulterated Rock Candy Syrup. Bought of E. Bradford Clarke Co.
2.	3244	Finley Acker Co., Philadelphia, Pa. Acker's "H. G." Golden Cane Syrup. Bought of Finley Acker Co., Philadelphia.
3.	3129	Abram Lyle & Son, Lyle's Golden Syrup.* Warranted free from Starch Glucose.
4.	3248	Wm. Montgomery & Co., Philadelphia, Maple Leaf Brand. Extra Fancy Pure Sugar Syrup. Guaranteed Absolutely Pure. Bought of John S. Marks, Phila- delphia.
5.	3187	Most Co., Inc., Pittsburg, Pa. Old Hickory Syrup.* Colored with Caramel and Flavored. Bought of A. S. Deiter, Reading, Pa.
6.	3242	Perfection Java Closure Co., Philadelphia, Pa. Perjanya Pure Golden Syrup. Made from Refiner's Syrup and Sugar. Highest quality of richness, flavor and purity and contains no corn glucose. It conforms to Pure Food requirements. Bought of Gimbel Bros., Philadelphia.
7.	3210	Rigney & Co., Brooklyn, N. Y. "Brand." Rock Candy Syrup.* Bought of W. A. Hartman, Sunbury, Pa.
8.	3223	A. E. Ritty Canning Co., Dayton, O. Ohio Syrup.* Pure and Deliciously Flavored. Bought of Geo. D. Williams, Eric, Pa.
9.	3132	J. Stromeyer & Co., Philadelphia, Pa. The Stromeyer Penn Mar Brands of Fancy Table Syrup. The Syrup is Warranted Purc, Wholesome and Delicious. Manufactured from Sclect Cane Sugar. Bought of Kreidler Bros., Harrisburg, Pa. Price, 20 c.

The analytical methods employed in these cases were in part like those used for maple sirups, in part like those used for cane sirups, according to the similarities they exhibited in physical properties. The analytical results were as follows:

REFINER'S MOLASSES, SUGAR SIRUPS, ETC.

6	3133 1.411 1.4865 26.66 22.44 1.76 21.20 79.20 36.96 38.14 3.289 7.745 0.066 53 11.3 11.3 11.3
00	1.327 1.4574 62.8 62.8 62.6 32.70 67.30 67.30 67.30 62.76 .019 .041 .041 .041 .041 .041
4-	2310 1.341 1.4506 54.0 -23.2 1.54 31.80 68.20 68.20 68.34 2.07 2.07 2.07 2.00 0.00 0.02 1.5 1.5 1.5 1.5 1.5 1.7 1.5 1.7 1.5 1.7 1.5 1.7 1.5 1.7 1.7 1.7 1.7 1.7 1.7 1.7 1.7 1.7 1.7
Ó	23.42 1.4538 1.4548 23.0 -18.0 5.3 21.55 78.45 30.91 40.40 2.55 40.50 2.32 2.32 2.32 2.32 2.32 1.14 1.14 1.74 1.74
rð	3187 1.334 1.4598 57.1 0.23 31.70 68.30 59.53 5.79 6.73 5.79 6.79 6.79 7.0 7.0 7.0 7.0 7.0
च्यं	2348 1.4873 1.4873 39.4 -10.2 2.6 2.6 2.6 2.6 7.940 7.7940
ŝ	3129 1.436 1.4904 20.8 20.8 23.8 4.0 19.40 89.60 83.62 40.66 1.83 4.59 1.74 1.74 1.74 1.74 1.39 1.3361
61	22.6 -23.6 -19.8 -19.8 -19.8 -19.8 -19.00 -1
н	3249 1.317 1.4583 56.4 -22.44 4.62 32.30 66.03 37.10 60.03 3.71 0.010 None. .03
Group Number	Agent's number, Specific gravity, Refractive index (28°C.), Diracted (20°C.), Indirect (20°C.), Indirect (20°C.), Solids (per cent.), Solids (per cent.), Sucrose (per cent.), Invert sugars (per cent.), Invert sugars (per cent.), Special Properties: Special Properties: Ash, insoluble (per cent.), Ash, per subject (20°C.), Insoluble (20

## Discussion of Analytical Results

None of these products responded to tests for benzoic and salicylic acids, nor for saccharin.

As respects solids, all met standard requirements within practical limits.

The golden sirups, 3244, 3129 and 3242, all show high percentages of invert sugar and less sucrose than the average amount found by Wallace in such sirups. The ashes in these sirups range from .73 to 2.52 per cent., smaller percentages than would be expected from the last of sweet water from the charcoal filter. These may be accounted for, in case of 3242 and possibly in other cases, by the adding of sugar solution to the refinery molasses; though the large proportion of invert sugar indicates that either the proportion of added sugar is small, or that it was inverted as part of the manufacturing process. Its general properties indicate that 3132 also belongs to this class of products. The high solubility of the ash is another characteristic of these sirups.

The rock candy sirups, 3249 and 3210, are marked by high percentages of sucrose and low percentages of invert sugar and ash.

Nos. 3187 and 3223 are flavored and colored sugar solutions.

The quantities of sulphur dioxide in the golden sirups are in all cases low, but in No. 3132, the only sample of this class in which zinc and tin were determined, the quantities fall within the range observed in case of molasses.

The low lead number and malic acid values for these products are noteworthy.

## Misbranding

The following points in which the labels appear misleading or otherwise not in conformity with the requirements of the law, are noted:

Labelled "Cane Syrup," but its lack of cane-flavor, its 3244:salty taste, and general composition indicate that is is not a cane sirup, but a refinery or sugar sirup.

Bears a device purporting to be a maple leaf, which suggests a maple product confrary to the fact in this case.

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## GLUCOSE, or "CORN SIRUP"

Glucose, as the term is used in American commerce, is a viscous liquid prepared by the partial hydrolysis of corn starch. The nature of the changes which starch undergoes in the process has been quite fully stated in the introductory pages.

T. B. Wagner<sup>1</sup>, of the Corn Products Refining Company, in a condensed description of the present mode of manufacture of this product, states that the starch separated from the corn by special processes, is suspended in a large volume of water containing a very little hydrochloric acid and is then heated under high pressure in tall, thick-walled copper converters until the starch is decomposed, the operation requiring about ten minutes. The liquid is then passed to neutralizing tanks where its acid is neutralized by the adition of soda ash, which acts upon the hydrochloric acid to form common salt, whose presence in small amount in the final product accounts for its slightly salty flavor. The neutralized liquid is then filtered to remove certain impurities, such as the fiber, etc., derived from the corn, and evaporated to a density of 30° Baume. The partially concentrated liquid is then decolorized by passing it through three successive bone-char filters, and finally condensed in vacuum pans to a density of 42° to 45° Baume.

The factory determinations of density are made, according to Gudeman<sup>2</sup>, at a temperature of about 140° F. and the results calculated to the equivalent specific gravity at 100°F; the temperature correction being 1° Baume additional for 40° F. decrease in temperature.

The standard proclaimed by the Secretary of Agriculture for this product is:3

"Glucose, mixing glucose, confectioner's glucose, is a thick, sirupy, colorless product made by incompletely hydrolyzing starch, or a starch-containing substance, and decolorizing and evaporating the product. It varies in density from forty-one (41) to forty-nve (45) degrees Baume at a temperature of 100° Fam. (37.7°C.), and conforms in density, within these limits, to the degree Baume it is claimed to show, and for a density of forty-one (41) degrees Baume contains not more than twenty-one (21) per cent. of water and for a density of forty-five (45) degrees not more than fourteen (14) per cent. of water. It contains on a basis of forty-one (41) degrees Baume not more than one (1) per cent. of ash, consisting chiefly of chlorids and sulphates."

This product is used not only in the preparation of "table sirups," but very extensively as a sugar substitute in the making of fruit products, such as jellies, jams, preserves and fruit butters, and of confectionery. When used as an ingredient of the latter classes of products it is usually called "glucose."

There has been an interesting discussion concerning the name this product should bear. Some food authorities have believed that the term "sirup" should be reserved for products conforming in origin and preparation to the national standard for sirup which has been stated on an earlier page, for the reasons that the best usage in food nomenclature has established this meaning for the term, that the interests of the people are best protected by a definite rather than a broad, loose usage of food names, and that the difference in the qualities of true sirups (consisting chiefly of sugars) and of mixing glucose is so pronounced as to make the discrimination in names highly useful to the careful buyer. On the other hand, the manufacturer has objected that the word "glucose" has been given so many undesirable associations in the minds of the general public as to make the compulsory use of that name for their product a hardship, has adduced a large volume of testimony from scientists to show that the word "sirnp" has been very broadly used, and has urged that, since there is no commercial corn sirup corresponding to the narrower definition of the words, the application of the name to the liquid glucose is not misleading; and finally, claimed that such use of the word "sirup" involved no more than its use in "refiners' sirup," which, by reason of established popular usage, had been recognized as a name for refiners' molasses. The Board of Regulation created by the National Food and Drugs Act of June 30, 1906, decided4 that it is lawful, under that Act, to label this product "corn sirup" and also that if to corn sirup there is added a small percentage of refiners' sirup, a product of the cane, the mixture is not mis-

branded if labeled "corn sirup with cane flavor." The practical ontcome of this decision has been the use of the generic term "sirup" for mixtures of glucose with small amounts of other saccabrin liquids, and the use of the qualifier "with cane flavor" for preparations whose flavor has no clearly recognizable resemblance to that of the sugar-cane itself, nor of sugar-cane sirup. If the buyer takes the precaution, however, to examine the finer print on the labels of such goods subjects of inter-state commerce, he will observe that, under the regulations governing the labeling of compounds, the "corn sirups" usually bear statements of the kinds and proportions of the principal ingredients.

Mixing glucose is a white, or faintly yellowish viscous liquid, of a consistence often described as "sirupy." In fact, however, the dextrin it contains makes its consistence very different from that of sirups proper, and causes it to resemble in this respect glycerine or very thickly mucilaginous liquids rather than sugar solutions. not readily fermentable and, in mixture with cane sugar products, retards the crystallization of the sugar. Its flavor is, however, so slightly sweet and otherwise so neutral that it has found no acceptance by the public as a table article, except in admixture with other substances that serve to favor the compound. The unmixed glucose dissolves rather slowly in cold water, much more readily in hot water.

Because of the fact of its use as an adulterant in many food products, there is probably much truth in the argument of the manufacturer that the consumer attaches many unpleasant notions to the word "glucose." As a matter of fact, the components of glucose, dextrose, maltose, and dextrin,—are recognized as nutritious foods. The National Academy of Science some years ago, affirmed its wholesomeness, and there is no objection, on the score of cleanliness, to the present methods of manufacture of the American product. Dextrose is one of the valuable sugars, in fruits and in cane sirup; according to Charrin and Brocard<sup>5</sup>, maltose responds more readily to the digestive fluids of the human body than do milk sugar and ordinary cane-sugar; while dextrin is one of the valuable constituents of every baked flour product.

Formerly, for the sake of securing a water-white product, bisulphite of soda was used in the last stages of the neutralizing process, and thus a little sulphur dioxide was introduced into the glucose. This ingredient had the effect of making glucose candy harden better. At the present time, however, the manufacturers, stimulated by prosecutions under the food laws of Pennsylvania and other States, appear to have eliminated this undesirable admixture, with the consequent removal of this ground of objection. Arsenic was reported, some years ago, in English glucoses made by the use of sulphuric acid prepared from pyrites; but it is said that pyrite products are not used in the American glucose factories, and recent analyses of the glucose on the American market show a commendable freedom from this impurity.

The composition of mixing glucose has not been at all frequently reported in full. Wagner' says that the average American product contains:

	Per cent.
Water,	. 19.0
Dextrose,	. 38.5
Dextrin,	. 42.0
Ash,	. 0.5
	100.0

But there is, in this statement, no mention of maltose and nitrogenous bodies, which are always present; nor of unfermentable condensation or reversion products, formerly called by the collective name, *gallisin*, which are probably always present in some degree.

Harrington<sup>6</sup> gives the analyses of five samples of unmixed glucose from the Chicago refinery as follows:

No.	Color	Water	Solids	Reducing sugars (Glucose)	Solids, not sweet
		%	%	%	%
5	Water white,	20.18	79.82	35.53	44.29
23	Water white,	19.20	80.80	41.15	39.65
24	Brownish red,	19.20	80.80	38,34	42.46
25	Yellowish,	19.90	80.10	38.34	41.70
28	Dark brown red,	18.90	81.10	38.34	42.76

All these samples were somewhat acid.

Leach<sup>7</sup> gives the following percentage range for the principal ingredients in glucose:

	70
Dextrin,	29.8 - 45.3
Maltose,	4.6 — 19.3
Dextrose,	
Ash,	
Water,	14.2 - 17.2

The writer has examined, in connection with the present investigation, five samples, taken from wholesaler's stocks, and marked "'Confectioners' glucose (43°B)" with results as follows:

Sample Mark.	A	3A	В	D	E
Specific gravity (60°F.),	1.445	1.455	1.433	1.440	1.435
	1.5023	1.4965	1.4988	1.4963	1.4964
Polarizations: Direct (20°C.), Indirect (20°C.), Indirect (87°C.),	166.4	180.8	167.4	173.8	175.6
	163.5	179.3	165.0	173.8	172.0
	151.8	173.8	159.1	162.4	165.9
Water (by difference, per cent.),	14.85	17.05	16.20	17.15	17.10
Solids (Geerlig's table, per cent.),	85.15	82.95	83.80	82.85	82.90
Reducing sugars as dextrose (per cent.)	39.35	32.06	38.34	36.72	35.24
Non-reducing organic solids (by difference, per cent.),Ash,	45.47 .33	50.41	45.06 .40	45.72 .41	47.31 .35
Percentage composition of organic solids: Reducing sugars, Non-reducing substances,	46.4	88.9	46.0	44.5	42.7
	53.6	61.1	54.0	55.5	57.3
Special Properties:  Ash, water soluble, per cent.,  Ash, water insoluble, per cent.,  Ash ratio, insoluble to soluble,	.23	.28	.30	.25	.29
	.10	.10	.10	.16	.06
	2.3	3.8	3.0	1.6	4.8
Ash alkalinity:  Water soluble (Cc.),  Water insoluble (Cc.),  Total (Cc.),	.26	.36	.34	.34	.36
	.22	.26	.22	.34	.18
	.48	.62	.56	.68	.54
Sulphuric acid, SO <sub>3</sub> in ash (per cent. in syrup), Chlorin in ash (per cent. in syrup),	.0137 .07	.0343 .06	.0206	.0686 .04	.0343
Corresponding percentage of com- mon salt,	.12	.10	.10	.07	.07

None of these samples gave off sulphur dioxide upon distillation with steam, and all were found free from saccharin and benzoic and salicylic acids.

The specific gravities, corrected to 100°F, by the subtraction of 1° Beaume from the equivalent of the specific gravities at 60°F, ranged from 42.8° to 44.35° Beaume. The readings were taken with a Westphal balance, but owing to the viscosities of these liquids at the temperature of observation (60°F.), it is believed that the method is much less delicate as an indication of the water and solids contents than is the determination of the refractive index, the applicability of the refactometer to this determination in glucose having been indicated by Bryan's results.

The polarizations of these samples at 20° C, before treatment for inversion by the Clerget method gave readings ranging from 166.4° to 180.8° Ventzke, average 172.8°. The readings at the same temperature after inversion, which took place upon standing at room temperature for 24 hours, after the acid addition, rather than by heating to 68° C, in fifteen minutes, showed decreases ranging from nothing to 3.6°, average 2.1°; and at 87° C, further decreases ranging from 5.5° to 11.7°, average 8.1° Ventzke; making the readings at

87° C. after inversion from 7.0° to 14.6°, average 10.2° Ventzke lower than those observed at 20° C. before inversion; so that the average readings after inversion were, at 20° C., 170.7°; at 87° C., 164.7° Ventzke. The decrease in rotation upon heating to 87° C. is not chiefly due to hydrolysis of the dextrin, for upon cooling again to 20° C., the same solution increases in reading to near the figure obtained after standing for 24 hours; and upon being again heated to 87° and re-cooled to 20° C., shows, at these temperatures, about the same degrees of activity as were first observed at the respective temperatures. These changes with the conditions of observation, must, in the case of mixtures with sucrose-containing products, affect the determinations of cane-sugar, and also those of invert sugar by the polarimetric method.

Leach<sup>9</sup> proposed that the maximum polarization at 20° C., uninverted, be accepted as 175° V.; his later investigations showed that 42° B. glucose made more recently polarizes as low as 162.4°V, and he judges that his former figure may be too high to represent the mixing glucose now in use for admixture with molasses, sirups, etc. It has been stated by various writers that glucose is sold of density varying with the purposes of its use; but Gudeman<sup>2</sup> declares that this is not the fact, but indicates that the variations in optical activity and in density are due to lack of entire control of the manufacturing operations. He affirms that the observed range of optical activity in goods of substantially the same density is too great to make practicable the use of Leach's suggestion even for an approximate ca'culation of the percentage of glucose in mixtures from their observed optical activity.

Leach's proposal has, however, been adopted by the Association of Official Agricultural Chemists, except in cases where much invert sugar is present, in which case the calculation is based upon the polarization at 87° C., and 163° V. is used as the glucose reading for that temperature.

The percentages of reducing sugars found have been calculated as dextrose. Since maltose has a considerably greater reducing power than dextrose, the computed percentages are doubtless somewhat too high, the excess varying with the quantities of maltose present in the glucoses.

The ash percentages are very small, and the ash is chiefly soluble in water, though composed in smaller degree than was anticipated, of chlorids and sulphates.

## Samples Analyzed

The official sampling agents submitted a relatively large number of samples whose labels declared, more or less plainly, that they contained "corn sirup." In some cases the main label contained the name "Corn sirup with cane flavor," in others the main name was simply "sirup" or "table sirup" with a more or less clear declaration that "corn sirup" formed part of the material, while in still other cases a declaration of the compound nature of the product was made or the names of the ingredients were combined to form the name of the mixture. All samples bearing such labels are described in the following list:

Group No.	Sample No.	
1.	. 3257	Alabama Georgia Syrup Co., Montgomery, Ala.: Alaga,* Alabama-Georgia Co's Cane and Corn Compound. A Blend of Pure Georgia Ribbon Cane Syrup and Corn Syrup. To keep same from Sugaring or Souring. Bought of the Mohican Co., Eastou, Pa. Price, 10 c.
2.	3217	M. H. Alexauder Co., Cincinnati, O. Dove Brand Refined Molasses and Corn Syrup. 65 % Pure Molasses, 35 % Pure Corn Syrup, thoroughly Refined. Con- tains Sulphur Dioxide. None Better for Any Purpose. Bought of J. Gray & Co., Williamsport, Pa.
3.	3234	Arbuckles' & Co., Pittsburg, Pa. Pond Lily Table Syrup. Extra Quality. Compound, 93 % corn syrup, 10 % sugar syrup. Contains Sulphur Dioxide. Bought of M. Dwyer, Pittsburg, Pa.
4.	3246	Atlas Specialty Co., Inc., Richmond, Va. Silk Velvet Brand Corn Syrup. Maple Flavor. Smooth as the name indicates. 1/10 % benzoate of soda. 85 % corn syrup, 15 % Refiners' Syrup. Bought of Merrill & Hopper.
5.	3194	Central Molasses and Syrup Refinery, Philadelphia, Pa. Family Table Syrup. Compound of Corn and Refiners' Syrup. Bought of E. N. Michael, Reading, Pa.
ø.	3128	Central Molasses and Syrup Refinery, Philadelphia, Pa. Family Table Syrup.* Compound of Corn and Refiners' Syrup. Bought of H. J. Bracony, Harrisburg, Pa. Price, 10 c.
7.	3273	Central Molasses and Syrup Refinery, Philadelphia, Pa. Our Favorite Brand Syrup. Stamped "Compound, Table." A compound of the best pure sugar syrup and corn syrup by which the flavor is made smooth and sweet, and the body and character generally improved. Bought of J. B. Warren, Gettysburg, Pa. Price, 10 c.
8.	3284	Corn Products Manufacturing Co., Davenport, Iowa. Atlas Corn Syrup with Cane Flavor.* 90 % Corn Syrup, 10 % Refiners' Syrup. Compound. It is of the highest quality and prepared according to U. S. Standards. Bought of A. F. Clapper, Everett, Pa.
9.	3159	Corn Products Manufacturing Co., Davenport, Iowa. Golden Glory Corn Syrup with Cane Flavor.* Compound 90 % Corn Syrup, 10 % Refiners' Syrup. 2½ lbs. net weight. Bought of D. P. Lantz. Laneaster, Pa. Price, 12 c.
10.	3272	Corn Products Manufacturing Co., Davenport, Ia. Royal Coru Syrup.* Cane Flavored, Choice Quality. 2½ ths. net weight. 10 % Refiners' Syrup, 90 % Corn Syrup. Compound. Delicious, Wholesome, Pure. Bought of Wagner Bros., Lewistown, Pa. Price, 12 c.
11.	3127	Coru Products Manufacturing Co., Davenport, Ia. Rex Corn Syrup with Cane Flavor.* Compound, 90% Pure Corn Syrup, 10 % Choicest Refiners' Syrup. 2 fbs. net weight. Bought of D. Pollock, Harrisburg, Pa.

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Group No.	Sample No.	
12.	3200	Corn Product Manufacturing Co., Davenport, Ia. Universal Corn Syrup with Cane Flavor.* 90 % Corn Syrup, 10 % Refiners' Syrup. Compound. Its ingredients are of the highest quality and prepared according to U. S. Standards. Bought of J. J. Bobbin, Shenandoah, Pa.
13.	3274	Corn Products Refining Co., New York. Banquet Table Syrup.* Corn Syrup, 30 %. Refiners' Syrup, 10 %. 2 lbs. net weight. Bought of J. B. Urnians, Gettysburg, Pa.
14.	3254	Corn Products Refining Co., New York. Blue Knot Table Syrup. Corn Syrup, 90 %, Refiners' Syrup, 10 %. 2 hs. net weight. Bought of W. H. Schug, Easton, Pa.
15.	3212	Corn Products Refining Co., New York. Geo. Bubb & Son, Distributors, Williamsport, Pa. Haleeka Club Table Syrup. Corn Syrup, 90 %, Refiners' Syrup, 10 %. 2½ lbs. net weight. Pure and Wholesome. First Quality. Bought of Geo. J. Koons, Williamsport, Pa.
16.	3259	Corn Products Refining Co., New York. Drake & Co., Distributors, Easton, Pa. Golden Crown Table Syrup.* Corn Syrup 90 %, Refiners' Syrup 10 %. 2 hs. net weight. Fure and Wholesome. Bought of Caparaso & Co., Easton, Pa.
17.	3256	Corn Products Refining Co., New York. Drake & Co., Distributors. Golden Crown Fancy Table Syrup.* Pure and Wholesome. Compound. Refiners' Syrup 10 %, Corn Syrup, 90 %. Bought of W. H. Schug, Easton, Pa.
18.	3253	Corn Products Refining Co., New York. Geo. M. Dunlap Co., Distributors. Dunlap's Compound Table Syrup.* Corn Syrup, 90 %; Refiners' Syrup, 10%. 2½ ibs. net weight. Pure and Wholesome. For General Table Use. None Better. Geo. M. Dunlap & Co., Chester, Pa.
19.	3281	Corn Products Refining Co., New York. Eagle Corn Syrup with Corn Flavor.* Corn Syrup, 90 %, Refiners' Syrup, 10 %. Bought of Crabbe & Co., Hyndman, Pa. Price, 10 c.
20.	3153	Corn Products Refining Co., New York. Globe Brand Corn Syrup with Cane Flavor.* Corn Syrup 90 %, Refiners' Syrup 10 %. Tops the Globe. Pure, Wholesome. Bought of Mohican Co., Wilkes-Barre, Pa. Price, 10 e.
21	3255	Corn Products Refining Co., New York. Gold Ribbon Table Syyrup.* Corn Syrup, 90 %; Refiners' Syrup, 10 %. 3 lbs. net weight. Pure and Wholesome. Bought of W. Schug, Easton, Pa.
22	3135	Corn Products Refining Co., New York. Harrisburg Grocery and Produce Co., Harrisburg, Pa. (Distributors). Sweet Clover Brand Table Syrup.* Corn Syrup, 90 %; Refiners' Syrup, 10%. 2 ibs. net weight. Bought of Wm. Kimmel, Harrisburg, Pa. Price, 10 c.
23.	3161	Corn Products Refining Co., New York. "Kairomel Brand" Corn Syrup with Cane Flavor.* Pure and Wholesome. 90 % Corn Syrup; 10 % Refiners' Syrup. 22 tbs. net weight. Bought of Franklin Groeery, Lancaster, Pa. Price, 12 c.
24.	3134	Corn Products Refining Co., New York. Karo Corn Syrup with Cane Flavor.*  Pure and Wholesome. 85 % Corn Syrup, 15 % Refiners' Syrup. 2 lbs. net weight.  Bought of Kreidler Bros. Harrisburg, Pa. Price, 10 c.
25.		Corn Products Refining Co., New York. Karo Extra Quality.* To Meet a Demand for New Delicate Flavor and Color. 85 % Corn Syrup, 15 % Granulated Sugar Syrup. Vanilla Flavor. Bought of —————. Price, —.
26.	3184	Corn Products Refining Co., New York, by Manierre-Yoe Syrup Co., Chicago. Recves, Parvin & Co., Distributors, Philadelphia, Pa. White Clover Brand Table Syrup. Corn Syrup 90 %, Refiners' Syrup, 10 %. 2½ hs. net weight. Pure and Wholesome. First Quality. Bought of Bentley & Kurtz, Reading, Pa.
27.	3280	Corn Products Refining Co., New York. Santec Brand Corn Syrup with Cane Flavor.* Corn Syrup 90 %, Refiners' Syrup 10 %. Two Pounds net Weight. Bought of Crabbe & Co., Hyndman, Pa. Price, 10 c.
28.	3136	Corn Products Refining Co., New York. White Flake Syrup.* Corn Syrup. 85 %. Granulated Sugar Syrup 15 %. 2 lbs. net weight. Bought of New York Groeery, Selinsgrove, Pa. Price, 10 c.
29.	3237	P. Duff & Son, Pittsburg, Pa. Duff's Fancy Table Syrup. Compound. 80 % Pure Corn Syrup, 20 % Pure Cane Syrup. Bought of A. Serafinn, Pittsburg, Pa.

Croup No.	Sample No.	
30.	3236	P. Duif & Sons, Pittsburg, Pa. Duff's Syrup. Compound. 80 % Pure Corn Syrup, 20 % Pure Cane Syrup. Contains Sulphur Dioxide. Bought of Mrs. B. Aaron, Pittsburg, Pa.
31.	3141	Evans-Burtnett Co., Harrisburg, Pa. New Century Compound Fancy Table Syrup. Bought of R. J. Peter & Son, Harrisburg, Pa. Price, 10 c.
32.	3144	Great Atlantic and Pacific Tea Co., Inc., Jersey City, N. J. A. & P. Choice Table Syrup.* Compound. Corn Syrup 90 %, Refiners' Syrup 10 %. Bought of A. & P. Tea Co., Scranton, Pa. Price, 10 c.
33.	3196	Hooven Mercantile Co., Reading, Pa. Heron Table Syrup. Compound. Corn and Refiners' Syrup. Superior Quality. Bought of J. W. Fenstermacher, Reading, Pa.
34.	3182	Hooven Mercantile Co., Reading, Pa. Sunbeam Table Syrup. Superior Quality. Compound. Corn and Refiners' Syrup. Superior Quality. Bought of J. H. Cassel, Reading, Pa.
35.	3145	Hudson Mfg. Co., New York. The Southern Molasses Co., Distributors, New York. B. & O. Brand. N. O. Molasses and Corn Syrup.* Will give that rich yellow color so much desired by good cooks in brown bread, ginger snaps, etc. Contains Sulphur Dioxide. Bought of A. & P. Tea Co., Scranton, Pa.
36.	3235	James A. McAteer & Sons, Inc., Pittsburg, Pa. Liberty Brand Compound Molasses and Corn Syrup. Contains Sulphur Dioxide. Bought of Orazio & Bro., Pittsburg, Pa.
37.	3283	Manierre-Yoe Company, Chicago, Distributors: American Beauty Brand Corn Syrup with Cane Flavor.* Corn Syrup 90 %, Refiners' Syrup 10 %. 2½ lbs. net weight. Bought at Everett, Pa. Price, 10 c.
38.	3263	S. Scheiver & Co., Easton, Pa. Vanilla Drips Brand Table Syrup. A Compound Cane Syrup and Glucose. Bought of J. Schuyler & Co., Easton, Pa.
39.	3183	Star Light Molasses Company, Philadelphia, Pa. Fancy N. O. Molasses Star Light Brand. Compound. Guaranteed to Bake. Bought of J. H. Cassel, Reading, Pa.
40.	3267	Steuart, Knatz & Co., Baltimore, Md. Golden Crown Syrup.* 60 % Corn Syrup, 40 % Sugar Refinery Syrup. Bought of G. W. Stewart, York, Pa. Price, 10 c.
41.	3133	Steuart, Knatz & Co., Baltimore, Md. Golden Crown Fancy Table Syrup.* A Blend of 40 % Sugar Refining Syrup and 60 % Corn Syrup. Bought of Kreidler Bros., Harrisburg, Pa. Price, 12 c.

Samples marked \* bore numbers of guarantees filed with the Secretary of Agriculture of the United States.

#### METHODS OF ANALYSIS

The methods of analysis employed in case of these mixtures were the same as for the preceding groups. They serve only indirectly, however, for an estimation of the proportions of glucose present and for an indication of the quality of the saccharin liquids used to impart flavor to the chief ingredient of the mixture, glucose.

It will be recalled that, under our conditions of inversion, all but one of the samples of confectioner's glucose polarized slightly less after inversion than before, the temperatures of observation being 20° C. at both times, and that the average depression was 2.1° V. In the tables following, the quantities of cane-sugar present have been computed by the usual Clerget formula, which assumes that

the decrease by inversion is wholly due to the conversion of the canesugar, which polarizes to the right, into invert sugar, which polarizes to the left. If it be assumed, however, that the glucose in these mixtures was depressed by inversion in like manner with the pure glucose, part of the change in activity observed after inversion must be ascribed to the glucose. The result, in case of the average of the 90: 10 mixtures, would be to decrease by about 1.3 per cent the cane-sugar percentage stated in the tables.

In calculating the proportion of glucose present, the official formula<sup>10</sup> prescribed for use with polarization at 87° C. is

The divisor being the average polarization, under these conditions, of glucose which polarizes at  $175^{\circ}$  V. before inversion and at  $20^{\circ}$  C.

ANALYSES OF "CORN SIRUPS" AND GLUCOSE COMPOUNDS

	Composition
	roperties and General Chemical Com
	General
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TO THE PERSON TO PERSON TO THE	Properties
2	. Physical P
	Phys
TALVES	I.

	% % Agent's number	Flavoring Ingredient Named on Ribbon cane syrup,Refined molasses,	Label.	Per cent. glucose claimed	Specific gravity	Refractive index. (28°C)	Polariff Sec. 30°C. Polariff Sec. 30°C.	20°0°. Signation (V°). Signati	D°78 H97al Seg & s	28.28 3.33 3.83 3.63 3.64 3.64 3.64 3.64 3.64 3.64 3.6	sbito8 % E & & & & & & & & & & & & & & & & & &	Gane-sugar (Clerget form	88.88 Reducing sugar as dex	Signary Dextrin, etc., by differ ence	(151. % . 4 . 57. 57. 58. 89. 68. 68. 68. 68. 68. 68. 68. 68. 68. 68	88.1÷(,O°78) & theoset & (87°C, )÷1.68
Refiners' syrup,         90         1.4768         1.22.0         105.6         11.87         26.35         77.65         13.87         26.38         34.58         2.52           Refiners' syrup,         90         1.300         1.4768         138.2         122.0         109.5         1.76.25         1.88         37.7         7.625         1.88         34.74         37.7         86           Refiners' syrup,         90         1.404         1.4814         146.8         137.7         138.2         22.80         77.10         4.89         35.74         40.22         39           Refiners' syrup,         90         1.404         1.4814         146.8         138.2         122.0         77.10         4.89         35.74         40.22         39           Refiners' syrup,         90         1.404         1.4814         146.8         140.4         148.5         146.5         146.5         146.5         146.5         146.5         146.5         146.5         146.5         146.5         146.5         146.5         146.5         146.5         148.5         146.5         146.5         146.5         146.5         146.5         146.5         146.5         146.5         146.5         146.5         146.5<	3234 3246 3194			 88°°	1.402 1.422 1.404	1.4872 1.4799 1.4823	138.0 138.0 134.6	128.5 144.3 127.6 123.4	138.6 125.4 122.3	23.50 22.55 22.15	77.45	9.57 7.84 8.44	27.52 32.74 33.14	38.86 35.62 35.07	1.25	75.0
Refiners' syrup,         9         1.383         1.4510         1.383         1.28.80         77.20         2.34         33.74         40.22         39           Refiners' syrup,         90         1.494         1.4814         146.8         138.2         22.80         77.20         4.89         35.74         40.22         39         1.48           Refiners' syrup,         90         1.385         1.454         146.0         118.6         138.2         25.36         74.65         1.81         30.30         40.90         1.64           Refiners' syrup,         90         1.887         1.473         130.8         121.0         119.0         24.55         77.45         7.31         30.3         1.04           Refiners' syrup,         90         1.887         1.473         132.0         127.6         1.81         30.3         1.40           Refiners' syrup,         90         1.481         149.0         122.1         120.5         77.45         1.53         1.65           Refiners' syrup,         90         1.481         1.474         136.4         128.7         129.2         23.46         33.64         1.38           Refiners' syrup,         90         1.487         1.4	3138 3273 3284	Refiners's Sugar syl Refiners'		-~-88	1.390	1.4828	122.0	103.6 135.7 132.0	107.8 130.9 128.5	22.35 24.75 23.75	77.65 75.25 76.25	13.87 1.88 1.66	26.88 34.74 33.06	34.38 37.77 40.91	20.2 98.0 20.0 20.0	8.08.08 18.08.08
Refiners' Syuth,         90         1.3857         1.4857         1.48.0         1.48.1         22.0         7.5.4         2.5.5         2.5.5         2.5.6         7.4.65         7.30         1.6.5	3159 3272	Refiners' Refiners'		3~8	1.393	1.4816	140.8	137.7	133.3	22.80	77.10	48.34	33.74 35.76 97.94	40.22 35.49 45.33	1.03	8.25.8 8.8.8
Refiners         Syrup,         90         1.385         1.4798         139.0         127.6         23.55         76.45         5.35         31.45         35.71         1.05           Refiners         Syrup,         90         1.400         1.4535         1.4753         135.8         135.8         35.71         1.05           Refiners         Syrup,         90         1.4857         1.4754         136.4         128.7         128.7         1.495         22.05         77.35         4.98         35.7         74           Refiners         Syrup,         90         1.4764         136.4         128.7         128.7         25.05         77.35         4.98         35.70         77.4           Refiners         Syrup,         90         1.4764         136.4         128.7         129.0         77.35         2.34         35.8         36.6         77.4           Refiners         Syrup,         90         1.4787         1.4704         132.7         127.8         27.05         1.93         1.40         1.40.4           Refiners         Syrup,         90         1.4873         1.40.4         132.7         127.8         27.05         4.98         35.25         76.55 <t< td=""><td>3200 3274</td><td>Refiners' Refiners'</td><td></td><td>888</td><td>1.385</td><td>1.4837 1.4754 1.4773</td><td>154.2 145.0 130.8</td><td>148.5 142.6 121.0</td><td>144.1 138.2 119.0</td><td>25.35 24.55</td><td>74.65</td><td>7.39</td><td>30.33</td><td>40.90 34.97</td><td>1.64</td><td>73.0 73.0 8.00</td></t<>	3200 3274	Refiners' Refiners'		888	1.385	1.4837 1.4754 1.4773	154.2 145.0 130.8	148.5 142.6 121.0	144.1 138.2 119.0	25.35 24.55	74.65	7.39	30.33	40.90 34.97	1.64	73.0 73.0 8.00
Refiners' syrup,       Syrup,       1.400       1.455.0       1.474.1       136.4       128.7       125.70       74.30       5.65       32.48       35.82       .95         Refiners' syrup,       90       1.378       1.474.1       136.4       128.7       127.6       25.05       74.95       2.34       35.38       .95       .95         Refiners' syrup,       90       1.412       1.4573       120.0       104.1       110.0       20.60       79.40       11.99       32.46       35.61       1.38         Refiners' syrup,       90       1.412       1.4573       120.0       104.1       110.0       20.60       79.40       11.99       32.46       35.61       1.38         Refiners' syrup,       90       1.4794       140.0       132.0       177.8       23.10       40.66       1.09         Refiners' syrup,       90       1.389       1.4806       150.0       143.4       145.6       23.25       76.50       4.98       35.92       35.19       37.19         Refiners' syrup,       90       1.388       1.4806       140.8       134.2       185.7       7.16       33.82       35.02       35.19       31.9         Refiners' syrup, </td <td>3212 3212 5959</td> <td>Refiners' Refiners' Refiners'</td> <td></td> <td>88</td> <td>1.385</td> <td>1.4798</td> <td>135.8</td> <td>132.0</td> <td>127.6</td> <td>23.55 25.35</td> <td>76.45</td> <td>7.31 4.98</td> <td>32.58 35.58</td> <td>38.71</td> <td>1.05</td> <td>55.6</td>	3212 3212 5959	Refiners' Refiners' Refiners'		88	1.385	1.4798	135.8	132.0	127.6	23.55 25.35	76.45	7.31 4.98	32.58 35.58	38.71	1.05	55.6
Refiners syrup,     90     1.389     1.4704     100.1     100.0     20.60     79.40     11.99     32.46     33.64     1.32       Refiners syrup,     90     1.389     1.4506     140.0     132.0     127.8     23.70     76.30     6.05     29.10     40.06     1.09       Refiners syrup,     90     1.389     1.4806     150.0     143.4     145.6     23.25     76.75     4.98     32.92     55.19     40.06     1.09       Refiners syrup,     90     1.389     1.4806     140.8     134.2     128.7     23.10     40.8     35.92     55.19     40.8     35.92     55.19     31.9       Refiners syrup,     90     1.388     1.4806     140.8     134.2     128.7     7.16     33.82     32.08     11.9	3256	Refiners' Refiners'		888	1.400	1.4744	136.4	128.7	123.2	25.70	74.30	5.05	32.48 32.98	35.82	60.00	75.6
Refiners' syrup,     90     1.383     1.4806     150.0     143.4     145.6     28.25     76.75     4.98     32.26     38.74     .77       Refiners' syrup,     90     1.388     1.4806     160.0     143.4     145.6     28.10     76.90     4.98     35.92     35.19     .81       Refiners' syrup,     90     1.388     1.4749     140.8     134.2     128.7     28.10     76.90     4.98     35.92     35.19     .81       Refiners' syrup,     90     1.383     1.4743     125.0     115.5     116.6     25.75     74.25     7.16     33.82     32.08     1.19	3281 3153	Refiners' Refiners'		388	1.412	1.4873	120.0	104.1	110.0	20.60	79.40	11.99	32.46 29.10	33.64 40.06	1.09	18.3
	3255 3135 3161	Refiners' Refiners' Refiners'	1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	3888	1.393	1.4806	150.0 140.8 125.0	143.4 134.2 115.5	145.6 128.7 116.6	23.25 23.10 25.75	76.75 76.90 74.25	4.98 4.98 7.16	32.36 33.92 33.92	38.74 35.19		86.57 8.0.61 8.0.61

† a = Polarization reading.

ANALYSES OF "CORN SIRUPS" AND GLUCOSE COMPOUNDS-Continued I. Physical Properties and General Chemical Composition

11		
89. I÷	Glucosc† a (\$7°C.)-	%88.88.88.65.7.08.88.66.7.7.08.88.66.7.7.08.88.7.7.0.6.6.7.7.0.6.6.7.7.0.6.6.7.0.0.0.0
	dsA	7,0 1.20 1.20 1.31 1.31 1.31 1.32 1.33 1.33 1.34 1.35 1.35 1.35 1.35 1.35 1.35 1.35 1.35
differ-	Deztrin, ete., by	28.39 28.39 28.39 28.39 28.39 28.39 28.39 28.39 28.39 28.39 29.36 38.39 29.39 38.30 38.30
-gez-	sa ragus guionbed escri	28.1.26 29.1.26 29.1.26 29.1.26 20.1
-mroi	Cane-sugar (Clerget	% 4.90 1.13 7.46 10.73 6.03 16.73 4.45 11.46 11.46 11.46 25.18 25.18 25.18 27.54 11.35 11.35 11.35 11.35
	spiloS	73.35 73.35 73.35 73.35 73.35 73.35 73.35 73.45 73.45 73.45 73.40 73.40
	Moisture	20.65 20.65 20.10
(A.)	Invert 87°C.	135.0 136.4 136.4 136.4 107.8 107.8 107.8 115.4 115.4 129.8 129.8 129.8 129.8 129.8 129.8 129.8 129.8 129.8 129.8
Palarization (V°)	Invert 20°C,	134.6 1146.1 1146.1 117.4 117.4 117.6 117.6 117.6 117.6 117.8 117.
Pala	Direct 20°C.	141.4 147.6 153.8 192.4 192.4 110.2 131.8 131.8 131.8 143.2 160.2 175.2 167.0
(58°C)	Refractive index.	1.4872 1.4734 1.4813 1.4774 1.4823 1.4765 1.4765 1.4765 1.4765 1.4765 1.4765 1.4781 1.
	Specific gravity	1.383 1.389 1.399 1.400 1.400 1.420 1.397 1.420 1.395 1.420
bəmist	Per cent. glucose c	38888
	Flavoring Ingredient Named on Label.	Refiners' syrup, Refiners' syrup, Granulated sugar syrup, Gran syrup, Cane syrup, Refiners' syrup, Rofiners' syrup, N. O. molasses, Refiners' syrup, No. molasses, Refiners' syrup, Corn syrup, N. O. molasses, Refiners' syrup, Refiners' syrup, Refiners' syrup, Refiners' syrup,
	Agent's number	3184 3280 3287 3287 3186 3196 3195 3195 3285 3285 3285 3285 3288 3288 3288 328
	Group number	62 52 52 52 52 52 52 52 52 52 52 52 52 52

# ANALYSES OF "CORN SIRUPS" AND GLUCOSE COMPOUNDS H. Special Chemical Characters

		ASI	н	Soluble ÷ In-		ivalent	8	$SO_2$
Group Number	Agent's number	Water soluble	Water insoluble	Ash ratio: Solub soluble	Chlorin	Sodium chlorid equivalent	Sulphuric acid, SO3	Sulphur dioxide, S
	-	%	%		%	%	%	P.p.m
,	3257	.67	.27	2.5			.0857	23
	3217	3.656	.91	4.0	17	.28	.0343	3
	3234	.69	.20	3.5 5.9	.17	.30	.0110	
	3246	.47	.08	3.8	.26	.43	.0398	
	3194 3138	.99 1.00	.18	5.6	.25	.41	.0343	
	3273	2.26	.26	8.7			.0343	1
	3284	.72	.14	5.1	.09	.15	.0151	
	3159	.45	.17	2.6	.08	.13	.0617	١.
	3272	.85	.05	17.0	.20	.33	.0343	
	3127	.87	.16	5.4	.29	.48	.1125 $.0782$	· ·
	3200	.98	.16	6.1	.19	.38	.0482	
	3274	1.32	.32	$\frac{4.1}{2.8}$	.16	.26	.0384	
,	3254 3212	.64	.22	4.0	.26	.43	.0960	
	3259	.80	.25	3.2	.17	.28	.0548	
	3256	.61	.13	4.7	.16	.26	.0480	
,	3253	.79	.16	4.9	.23	.38	.0755	
	3281	.79	.14	5.6	.19	.31	.0411	
	3153	1.01	.31	3.3	.29	.48	.0302	
,,	3255	.90	.15	6.0	.11	.18	.0247	1
	3135	.56	.21	2.7	.13	.21	.0412	
	3161	.70	.11	6.4 5.0	.26	.43	.0686	1
,	3134 K	.99	.11	2.8	.04	.07	.0453	1
	3184	1.04	.16	6.5	.25	.41	.0521	
,	3280	.65	.14	4.1	.11	.18	.0133	
,	3136	.46	.07	6.6	. 09	.15	.0206	
	3237	1.94	.22	8.8	.47	.78	.0110	
,	3236	1.11	.20	5.5	.23	.38	.0523	
,	3141	2.39	.41	5.4	0.1	40	.0720	
	3144	.87	.25	3.5 4.5	.24	1.07	.0480	
,	3196	$\frac{1.62}{1.08}$	.36	5.1	.30	.50	.0523	
,	$3182 \\ 3145$	1.08	.77	2.6	.00	.00	.3121	
	3235	.58	.19	3.1			.0480	
,	3283	.78	.13	6.0	.18	.30	.0261	
	3263	3.27	.70	4.9			.0617	
),	3183	3.70	.89	4.2			.2559	
),	3267	1.94	.26	7.5			.0652	
,	3133	1.95	.24	8.1	.40	.66	.0429	

# Discussion of Results

The first question naturally arising is concerning the truth of the label declarations as to the percentages of glucose in these mixtures. It will at once be noted that, except in the cases of Nos. 3246, 3200, 3256, 3135, K, 3280, 3136, 3236, and 3267, where the quantities stated agree substantially with those calculated by the formula used in the table, the quantities computed are quite remote from the label declarations; and that, except in the cases of No. 3133, where the quantity is greatly excessive, the percentages are far less

than the labels declare. If the kind of flavoring liquid admixed with the glucose is correctly stated, the consumer is probably little, if at all, injured by such over-statement.

This difference is so great, however, that it requires especial consideration to determine whether any cause, other than a deliberate diminution of the proportion of glucose in the mixture, can account for it. The glucose average for the twenty samples for which 90 per cent. of "corn sirup" is stated on the respective labels, is 80.10 per cent., if we take the tabulated computations. If the calculations be based upon Leach's formula for use with the direct polarization at 20° C., adopting 175° as the maximum polarization of commercial glucose at that temperature, the average for these "90 per cent." samples is 77.30 per cent.; or, if we correct approximately the cane-sugar figures for the average decrease in optical activity upon inversion observed in the case of the confectioners' glucose we have analyzed and a proportion of 80 per cent. of glucose in the mixture, the average computed by this corrected method is 78.06 per cent.

A dilution of the mixture with water would tend to lower the polarizations and thus account, in part as least, for the observed depression. The confectioners' glucose we examined contained \$3.53 per cent. of solids on the average; but a minimum of 79 per cent. is recognized as occasional in mixing glucose. For refiners' sirup, a minimum of 75 per cent. is recognized as allowable, though 79 per cent. is more nearly the average; while sugar sirups may contain as low as 65 per cent of solids. Disregarding the one case in which "sugar sirup" is the declared flavoring material, in the twenty cases for which 90 per cent. of "corn sirup" is claimed, the minimum of solids present should be 78.6 per cent., whereas the amount found was 76.39 per cent., the quantities being less than the fheoretical minimum in eighteen out of the twenty cases; the average depression below the average for the respective ingredients represents the equivalent of 5 per cent. addition of water, either direct or indirect.

The average of the organic solids not sugar in these twenty cases, was 38.09 per cent.; if these be attributed to the glucose, thus disregarding the fraction of one per cent contributed by the refiners' sirup, the quantity would correspond to 47.60 per cent. of the glucose, if 80 per cent. be assumed as the average amount present; to 42.31 per cent. of the glucose, if 90 per cent. be regarded as the proportion of glucose present and to 44.80 per cent. for 85 per cent. of the glucose. The average percentage found in the confectioners' glucose we analyzed was 47.39 per cent.

The quantity of organic solids not sugar present in the average mixture is, in other words, what we should expect, if 80 per cent of confectioner's glucose, such as we have analyzed, were used in making

the mixture. Attention has already been directed to the limitations of the analytical data obtained as they apply to the computations of organic solids not sugar. For this reason, the comparison just made is merely suggestive, not at all conclusive.

It remains to be observed that the computation of the glucose proportions stated in the table is based upon the assumption that the optical activity of the glucose used was equivalent to 163° V at 87° C. after inversion, or about 175° V at 20° C. before inversion, these being the officially accepted figures. If the hydrolysis be carried a little farther, in one case than another, the dextrin, which is highly dextro-rotatory, will be more completely converted to maltose and dextrose, which are much less optically active, although dextro-rotatory. The consequence is that, without any decrease in the final quantity of solids, the optical activity of more the completely hydrolyzed glucose is reduced, possibly to a considerable degree.

Confining the calculations to the range of polarization at 87° C. observed in the confectioners' glucose samples that we analyzed in connection with this investigation, and making no correction for differences in the solid contents of the mixtures and glucoses respectively, the polariscopic reading of 130.56° V the average for the 90:10 mixtures, would correspond to 75 per cent or 86 per cent of glucose, according as 150° or 174° V, the respective extremes of the polarizations, invert, at 87° C., noted in our glucose analyses, were used for the computation.

Owing to the variable nature of the flavoring admixtures, there is nothing in the analytical data obtained in the present series of analyses, to prove the real degree of hydrolysis of the average glucose mixture under consideration; and, therefore, aside from the low solids in the mixtures, no established explanation for the low proportions of glucose indicated by the formula here employed.

The second question which arises is concerning the truth of the label statements concerning the nature of the flavoring liquids used in the mixtures and compounds.

In two cases, Group Nos. 25 and 28, sirup prepared from granulated sugar is claimed. The low ash, and, in case of No. 25, the high cane sugar content, tend to support this claim. In the case, however, of Group No. 7, the high ash is incompatible with the claim that the flavoring ingredient is "sugar sirup" in the true sense of the term, which means a solution made by dissolving sugar. Instead, the material was a substance relatively high in ash.

In 29 cases out of the 41 analyzed, the flavoring ingredient claimed is refiners' sirup. The ash in these cases ranges from .55 to 2.20 per cent, and averages 1.00 per cent. In the 19 cases in which the proportion of 90 parts of glucose to 10 of refiners' sirup is claimed, the ash range was .62 to 1.64; average 1.00 per cent. Taking 0.4 per

cent of ash as the normal for the glucose, this would give us averages of 3.2 to 7.1 per cent of ash, according as we accept 20 or 10 per cent as the proportion of refiners' sirup in the mixture; both these percentages falling, however, within the range for refiners' sirup. low figures for cane-sugar in Group Nos. 9, 13, and 27 indicate for these mixtures the use of sugar products of highly inverted character, and probably but not certainly low quality; the high ash in No. 13 points in the same direction. Owing to the variability in the proportions of sulphates found in the ash of unmixed glucose, the quantities of this constituent found in the several mixtures throw little light upon the quality of the flavoring material. On the other hand, the quantity and solubility of the ash in refinery molasses, are variable, as contrasted with these properties in glucose, but have not yet been correlated so definitely with the various grades of these refinery products, except in cases where the ash is extremely high, as to make them especially valuable for present judgment of the quality of the flavoring material in these mixtures.

The flavors of the mixtures having the names of the same ingredients were often quite unlike, especially when sugar sirup, cane sirup and refiners' sirup were the flavoring materials. In a few of the cases in which the latter was claimed, there was a slight, molasses-like flavor; in a few cases, a pronounced "butter scotch" flavor; but in the remainder, the flavor, where not affected by fermentation, was not readily likened to that of any simple material. Where large quantities of molasses were claimed, the flavor indicated the presence of this ingredient.

The quantities of sulphur dioxide found in these mixtures were, as a rule, very small, one-fifth of the samples showing none at all. It will be recalled that the legal tolerance for this added constituent covers only molasses, among these sngar products; that neither canesirup nor glucose comes within the protection of the proviso in which the tolerance is expressed; and that, since refining sirup also is omitted from mention, it is reasonable to regard the presence of sulphur dioxide in this refiners' product as constituting an adulteration. The same processes which led to its tolerated presence in molasses may have affected the raw sugars also, and thus led to its later appearance in the simps constituting the secondary products of the refining operation. The writer is acquainted with no exact studies upon the question as to what amounts, if any, of sulphur dioxide are thus carried forward through the refinery from the sugar-house.

We note that Group No. 25, said to be a mixture of glucose and granulated sugar sirup, contains the highest amount, save one, of sulphur dioxide; since this is not contributed by granulated sugar, it must have entered with the glucose; yet No. 24, which bears the

same brand name, but claims refiners' sirup as the flavoring ingredient, contains less than one-ninth as much of the sulphur dioxide. Group Nos. 10 and 40 also show more of this prohibited constituent than can be satisfactorily accounted for by the refinery products said to be used for the flavoring of the mixture.

The quantities apparent in Group Nos. 2, 35, 36, and 39, may be due to the molasses present; but, if so, it is to be noted that No. 39 fails to bear upon its label the required declaration of the pres-

ence of sulphur dioxide.

In the case of No. 3, such a declaration is made, but neither glucose

nor sugar sirup should contain it.

Although these products are commonly sold in tin cans, the original plan of the investigation included the determination of tin and zinc in the case only of the cane-sirup and molasses. It is possible to make no statement, therefore, as to the presence or absence of these metals from these glucose mixtures.

Tests for saccharin and the preservative acids, benzoic and sali-

cylic, gave negative results in all cases.

# Misbranding

In addition to the provisions of the Food Act of May 13, 1909, that prohibit the use of false, deceptive, or misleading names, statements, designs or devices upon food labels, those provisions, also, requiring the statement, in case of food mixtures, of the mixed or compound nature of the article, and of the substance entering therein, and finally the provisions relative to the place where and size of type whereby these required declarations shall be used, apply to these glucose compounds. The law requires that all labeling required by the Act shall be on the main label, in type not less than 8-POINT BREVIER CAPS, (unless the size of the package will not permit so large type), and in such position and terms as shall be plainly seen and read by the purchaser. For the purposes of this report, it is assumed that such position is upon the front part of the label, i. e. that part containing the brand name, which is usually made visible on the retailers' shelves.

It may first be noted that the meanings of the words "corn sirup" and "with cane flavor" have not been interpreted authoritatively as they apply in the purely internal commerce of the State.

The following sets of notes sufficiently indicate the salient defects in the labels, as gauged by the law's requirements:

#### DECLARATION OF NATURE

Group No.	
3.	Front of label does not indicate mixed nature; the word "compound" and statement of the ingredients and of the presence of sulphur dioxide is made on the side in small type
5.	Mixed character not indicated on front of label; and "compound" and names of ingredicnts appear on the back in small type.
12.	Declaration of compound nature and of the presence of sulphur dioxide, appears on the side, not the front of the label. The brand name does not indicate that the materia is mixed or compounded.
14.	Name does not indicate its mixed or compound nature; statement of ingredients, while on front label, is in black type on a dark blue ground, and therefore difficultly legible.
17. 21.	Compound nature and name of ingredients on side of label.  Brand name does not show mixed nature. Names of ingredients in small type, black on red ground, on front label.
25.	Main label bears no indication of mixed nature, though the brand name does not give any indication of the nature of the article. The names of the ingredients appear on the sides of the can.
30.	Declaration of compound nature and of the presence of sulphur dioxide, appears on the side, not the front of the label. The brand name does not indicate that the material is mixed or compound.
31.	Names of ingredients omitted.
32.	Brand name does not show mixed nature. Word "compound" with names of ingredients in small type on the side of the can.
33.	The declaration of the compound nature and of the ingredients is placed in small type inconspicuously at the bottom of the main label.
34.	The declaration of the compound nature and of the ingredients is placed in small type inconspicuously at the bottom of the main label.
35.	Declaration of sulphur dioxide in small type and on the edge of the label.
36.	Presence of sulphur dioxide declared in small black letters on red ground, and on side of label.
39.	Word "compound" in very small white letters, in an inconspicuous position on the main label.
40.	Mixed nature not apparent in main name. Declaration of ingredients appears in very small letters at the bottom of the main label.
<b>£1.</b>	Mixed nature not apparent in main name. Declaration of ingredients appears in very small letters at the bottom of the main label.

#### MISLEADING STATEMENTS

2. Label declares "None better for any purpose". As a matter of first the first transfer of the first transfer

- 2. Label declares, "None better for any purpose." As a matter of fact, the flavoring quality of molasses and its degree of reaction with baking soda are weakened for baking purposes, by mixing practically neutral glucose with it.
- 4. | Maple flavor claimed. Could not taste it.
- 5. Bears device of girl with sugar-cane, no suggestion by device that at least three-fourths of the material is derived from corn.

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- Bears device of girl with sugar-cane, no suggestion by device that at least three-fourths of the material is derived from eorn.
- Label elaims that the glucose makes "the best pure sugar syrup" sweet.
- Label states, "It is prepared according to U. S. Standards." There is no such standard for a glucose mixture with refiners' syrup, but only for the ingredients taken separately.
- Labelled "Pure," but contains 32 p.p.m. of sulphur dioxide. 10.

It is clear that, in the particulars here discussed, the buyer must have much trouble in discovering the truth from the labels on many of these goods.

### NET WEIGHT OF PACKAGES

Upon the labels of this class of products, the net weight of the contents has been stated on a large fraction of the labels. weights have been determined in these cases, with the result that, in most cases, a slight excess of contents was observed; and in the few cases of deficiency, this amounted to more than one per cent. of the stated weight in but one case, when the deficiency was about 1.7 per cent of the stated weight. In general, therefore, the consumer has little ground for complaint of short weight with respect to any of the samples thus examined.

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#### ACKNOWLEDGMENTS

The efficient work of the assistants who performed the analytical work under the writer's direction, deserves mention. The determinations of refractive index, polarization, and reducing-sugars, were made by Luther G. Willis, B. S.; those of specific gravity, ash, chlorin and sulphuric acid, by H D. Edmiston; those of sulphur dioxide, lead number, malic acid value, tin and zinc, and also the tests for saccharin, benzoic and salicylic acids, by Walter Thomas, B. S. To Assistant Professor Given acknowledgment is due for many helpful suggestions.

#### SUMMARY

The examination of the table sirups on sale in Pennsylvania reveals the following conditions, with respect to the several classes of these products:

- 1. The goods sold as 'maple sirup' are, with few exceptions, true to name. Glucose has almost disappeared as an adulterant of these sirups. If ordinary cane sugar is used as an adulterant, it is within such limits that the characteristic constituents are not depressed below the range of variation they exhibit in authentic maple sirups.
- 2. The sirups labelled 'maple and cane' or 'cane and maple,' usually contain only small proportions of the maple product.
  - 3. There is little sugar-cane sirup on the market.
- 4. The molasses on sale is practically confined to the products of the modern sugar-house. The 'New Orleans molasses' of today differs materially in flavor, sugar-richness, and probably also in acidity from the older 'open-kettle' product formerly sold under that name. The ash limits for table molasses are exceeded in but few cases, but the organic solids not sugar are, in some cases, so high as to suggest that the molasses in these cases is of very inferior grade. The quantities of sulphur dioxide are usually not high. The presence of tin and zinc in small quantities is common, and, in the latter case, suggests the need for greater care in cleansing before filling the tins in which these goods are usually retailed.
- 5. The samples of refined molasses submitted were too few to permit any judgment of their average quality as compared with that of the ordinary molasses.
- 6. The 'corn sirup with cane flavor' and 'compound molasses' formed a large fraction of the 'table sirups' on sale. The goods of the former class frequently lack cane-flavor, in the literal sense of the term. The proportions of glucose claimed on the labels are rarely exceeded in the goods. As a class, these goods contain more water than should be introduced in the ingredients named. The proportions of sulphur dioxide present are, as a rule, very low, indicating that the glucose now used for these mixtures is practically free from this constituent, which was formerly found in larger amounts.
- 7. Sulphur dioxide appeared, in a few cases, in classes of sirups not covered by the proviso of the food law that tolerates it, within certain limits and under certain conditions, in molasses.

8. In no case was saccharin, benzoic acid, or salicylic acid found

present.

9. The labelling of many of these samples was open to objection, in that the indication of compound or mixed nature, the names of the ingredients of the mixtures, and the presence, in molasses, of sulphur dioxide, were often stated obscurely, and not plainly, in the position and size of type specified by law. Moreover, in mixture names, especially 'maple and cane', the less abundant ingredient was often made the more prominent, not simply by priority in order, but also by distinction in the typography used. In addition, some of the labels bore misleading statements or misleading devices. On the other hand, where weights of contents were stated, the quantities of contents corresponded closely thereto.



# **INDEX**

	Pag	æ.
	7	71
Achinard, P. E.,	E	52
Achinard, P. E.,	7	71
Allen, A. H.,	-	18
Allen, A. H., Amylase, in pancreatic juice,	81. 8	82
Analytical tables: (Pennsylvania samples) 38, 39, 44, 40, 61, 62, 66, 78,	61.6	$\bar{62}$
Analytical tables: (Pennsylvania samples) 38, 38, 11, 16, 42, cane sirup,	81,	82
cane sirup, corn sirup, glucose,	ĺ,	75
	81,	82
glucose, glucose compounds, golden sirup,		69
	38,	
maple sirup, maple sirup compound,	44,	
	61,	
		69
		69 CO
rock candy strups,sugar sirups,		69
sugar sirups,		34
Babbington,	-57,	
Babbington, Bagasse,		49
Bagasse, Bastards,		67
Bastards,		16
Barley sugar formation,		$\frac{71}{54}$
Beam, Wm., Beet molasses, qualities,		54 31
Beet molasses, qualities, sugar ash, composition,		ÐΙ
Black strap (See molasses),	64.	71
Rlouin R. E		•
Brocard, (See Charrin), 49, 50, 51, 53, 56, 64,	70.	71
Brocard, (See Charrin), 49, 50, 51, 53, 56, 64, Browne, C. A., 29, 31, 39,	40.	48
Browne, C. A.,	,	
		28
Calcium malate, in maple products,		72
Cane flavor decision on meaning of,		12
		52
		53
		52
manufacturing methods,		$\overline{52}$
open-kettle, sugar, action upon metals,		16
sugar, action upon metals,		31
		17
		16
		16
		31
		16
		14
		12
Carbohydrates, nature and rood value,		55
		73
		36
Clark, W. S., Clerget formula,		$-30 \\ -34$
		04
Color-test, maple sirup, Confectioners' glucose, (See Glucose), Cooke, W. W. and J. L. Hills,	27	7.48
Cooke, W. W. and J. L. Hills,		,
Corn sirup (See Glucose), for glucoses and glucose compounds. Po	enns	syl-
Vania samples,	3 1998	72

	Page
Dextrin, achro,	. 1.8
erythro,	. 17
general properties, Dextrose, digestion of,	. 18 . 14
fermentation,	. 14
fuel value,	14
multirotation,	. 14 . 14
d—Fructose. (See Levulose).	, 14
d—Glucose, (See Dextrose),	
Diastase, action upon starch, Di-saccharose,	$\begin{array}{cc} & 17 \\ 12 \end{array}$
2.2000401000,	1.2
Eaton, E. N.,	31.41.48
Edmiston, H. E.,	80
Edson, A. W., See Jones,	23,47,48
Folling's solution action upon sugars	1.
Fehling's solution, action upon sugars, Fermentation, inversion during,	$\begin{array}{cc} 14 \\ 15 \end{array}$
cane-sugar,	. 13
dextrose,	1.3
levulose, molasses, molasses,	$\begin{array}{cc} 14 \\ 57 \end{array}$
Fileti	65.71
Foam test, maple sirup,	34
Frear, William,	25,48,73
Fuel value of dextrose,	. 14
	J. J.
Gallisin,	74
Given, G. C.,	90
Gladstone, J. H., Glucose, in saliva and pancreatic juice,	$\begin{array}{c} -65,71\\ -18\end{array}$
Glucose, ash,	76
composition,	73
confectioners', gallisin in,	$\frac{74}{74}$
manufacture of,	$\frac{74}{71}$
mixtures, methods of analysis,	79
national standards for,	$\frac{72}{72}$
polarization,	73 75
present purity,	73
principal uses,	72
properties, reducing sugars in,	$\frac{73}{76}$
Glycogen,	14
Golden drips, composition,	68
Golden sirup, composition,	68
composition of Pennsylvania samples, definition, definition,	$\begin{array}{c} 70 \\ 67 \end{array}$
Grape sugar. (See Dextrose).	
Green sirup, composition,	68
definition, Gudemann, E	$\begin{array}{c} 67 \\ 76 \end{array}$
, and the state of	10
Hall, J. A., Jr.,	51,71
Hardin,	50
Harrington,	74 $19,48$
Hills, J. L.,	2.33.48
Hubbard, W. F.,	27,48
Invert sugar, optical activity,	15
sources and properties,	15,16
Iso-maltose, properties,	17
Jones, C. H.,	4,47,48
Jones, Edson and Morse	21.47

	Page.
Kreider, J. C., (See Winton, A. L.),	
Leach, A. E., Leach, A. E., and H. C. Lythgoe, 33	33,74 ,48,71
T 1	71
Lead number, (See Maple Strup), Leffmann, H., and W. Beam, Levulose, digestion of,	$1\overline{5}$
multipototion	$\bar{15}$
gourges and properties	14
Tintney and Dull	17
Lock	71
Lythgoe, H. C., (See Leach, A. E.),	
	0.0
McGill, A,	33
Malic acid value, (See maple sirup), Malt-sugar, source and properties,	17
Malt-sugar, source and properties,	
Maltase, (See Glucase), Maltose, (See Malt-sugar),	
Manager in monio Wood	28
NI - mm occo	28
Monlo concrete	$\begin{array}{c} 24 \\ 22 \end{array}$
Maple sap, albuminoids in, bacterial fermentations of,	$\frac{22}{23}$
flow, conditions of,	$\frac{20}{20}$
quentity	21
ice sugar in	23
malate of lime in	$\frac{22}{2}$
niter denosits composition	$\begin{array}{c} 22 \\ 28 \end{array}$
nitrogenous compounds of,	$\frac{20}{22}$
reducing sugars in, sugar richness,	$\frac{21}{21}$
Monlo girup adulteration kind and extent	28
methods of detection	$\frac{29}{2}$
aroma test for	34
ash, composition,	29,40
clarification, effect upon composition, compound, composition, relation to ingredients,	$\frac{30}{41}$
misbranding of, misbranding of,	47
Ponnsylvania samples composition,	41
low maple content	47
doterioration	$\frac{25}{25}$
distinctive qualities,	$\begin{array}{c} 25 \\ 24 \end{array}$
effects of filteration, general composition,	$\frac{23}{25}$
inverted, dextro-rotation,	39
lead number. Hortvet method,	32
McGill method,	33
Sy method,	$\frac{32}{32,37}$
Winton method,	32,31 32
lead precipitate from, malic acid value,	33,40
mothods of manufacture	23
Pennsylvania samples, composition,	34,39
regions of production	19
standard concentration,	$\substack{24,27\\26}$
Canadian,	
sugaring off,	
Sy color test for	34
Sy foam test for	34,40
species used for sugar,	$\frac{20}{29}$
sugar, ash composition,	
general composition,	
origin in tree,	19,27
standard national	. 26
wood starch in.	. 19
Mapleine	. 28
Mixing glucose, (See glucose), Misbranding, legal definitions,	. 66
Misbranding, legal definitions,	. 58
ash	. 63
beet,	$\frac{54}{}$
black stran	

	Page.
composition,	. 57
chlorides in, fermentation of,	57
first, second, and third, compositions,	57 56
general nature,	55
inverted, dextro-rotation,	63
lead in ,	. 65
Louisiana standard for, methods of manufacture,	. 63
mispranging,	66
National standard for.	57
New Orleans, composition,	56
official definition of,	. 58
old fashioned, Pennsylvania samples,	$\frac{55}{50}$
open kettle, composition,	$\begin{array}{c} 59 \\ 56 \end{array}$
method of manufacture,	54
Pennsylvania samples, composition,	58
quality,	62
Porto Rico, composition, refined, processes of manufacture,	56
Pennsylvania samples,	57
renners' (See Refiners' molasses).	•
sulphurous acid in,	64
tin in,	60
zinc in,	60
Monosaccharoses, Morse, W. J., (See Jones).	12
Multirotation,	14,15
	11,10
Newlands, Bros.,	71
Preservatives,	
Polarization,	39
	12
Reference lists, glucose,	89
maple products	47
sugar cane products	70
ttenners morasses, remisvivanta samples composition	68
misbranding,  Refiners' sirun national standard	70
Refiners' sirup, national standard, Refractive index, sugar solutions,	68
RUCK Candy Strup, compositions	13 68
manufacture,	67
Pennsylvania samples,	69
Saccharin,	39
Saccharose, (See cane-sugar). Sadtler, S. P	7.1
Salkowski,	$\frac{71}{65,71}$
Schroeder Julius,	1.30.48
Spaulding and White	19
Specific gravity of Sugar Solutions	13
Starch, digestion, hydrolysis	18
hydrolysis, soluble, formation,	$\begin{array}{c} 18 \\ 17 \end{array}$
sugar, (See Dextrose).	
Storer, F. H.,	8,47,48
Sucrase, in pancreatic juice,	18
Sucrase, (See Cane-sugar). Sugar cane, culture in America,	4.0
juice, acids,	$\frac{49}{51}$
ash,	$\frac{51}{51}$
general composition,	49
gummy bodies,	51
nitrogenous bodies,	50
repressing, composition, sugars,	51 49
milling,	49
products,	49

	Page.
sirup, (See Cane-sirup). National standard, Pennsylvania samples, refining processes, sirup, national standard, Pennsylvania samples, Sugars, classes of, Sulphitation, Sulphurous acid, investigation upon wholesomeness, use in sugar manufacture, Summary,	53 58 66 68 68 11 54 64 64
Summary,	2,04,40
Table sirups, American use, commercial sources, constituents, Thomas Walter, Treacle, (See also Refiners' sirup), composition, definition, Tin, determination in molasses, quantity in molasses,	11 11 11 90 67 68 68 67 60 65
Wagner, T. B., Wallace, Warren, W. H., Weber, H. A., Weights, net, White, (See Spaulding). Wiley, H. W., Willis, L. G., Winton, A. L.,	$\begin{array}{c} . & 22,48 \\ . & 65 \\ . & 89 \\ \hline 64,68,70 \\ . & 89 \\ . & 32,48 \end{array}$
Winton, A. L., Wood,	. 21,48
Zinc, determination in mclasses,	. 60

